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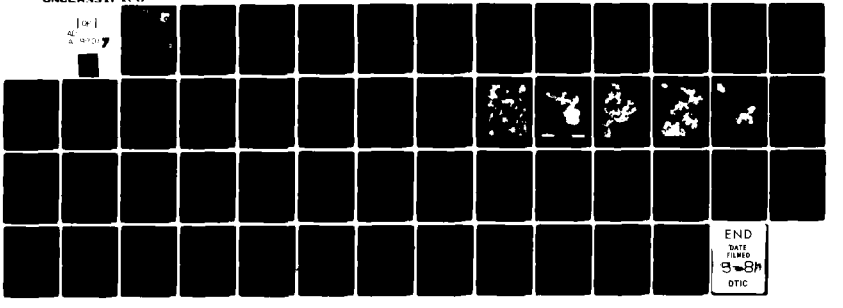

AFWAL-TR-80-2127

# CATALYST COMPLEXED CARBON SLURRY FUEL DEVELOPMENT

R. A. Johnson
R. L. Linard

Ashland Chemical Company
Specialty Fuels Department
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Dublin, Ohio 43017



January 1981

Technical Report AFWAL-TR-80-2127
Final Report for Period April 1980 – November 1980



AERO PROPULSION LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT–PATTERSON AIR FORCE BASE, OHIO 45433

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This technical report has been reviewed and is approved for publication.

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FOR THE COMMANDER

Robert D. Sherrill

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Turbine engine fuel
Catalyzed carbon slurry fuel
High density fuel
Carbon black

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

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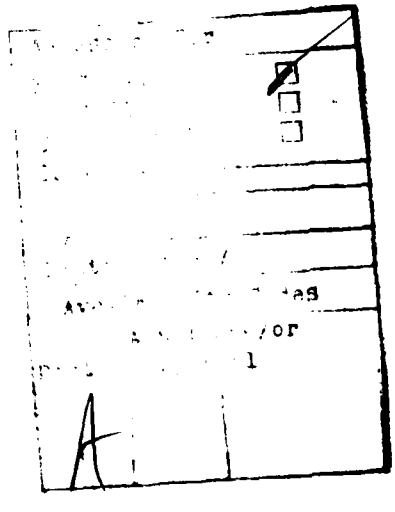

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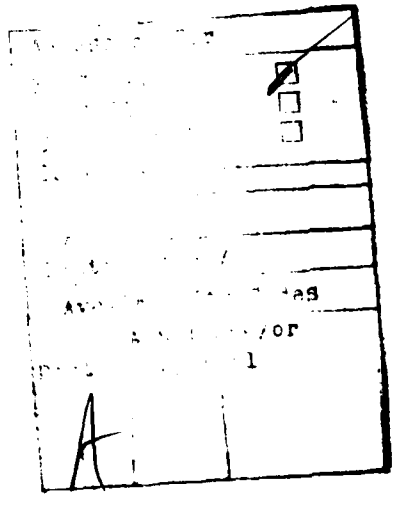

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|---|---|
| A/A | Measuring system for Contraves Viscometer |
| AMD | Arithmetic Mean Diameter |
| ANSI | American National Standards Institute |
| ASTM | American Society for Testing and Materials |
| B/B | Measuring System for Contraves Viscometer |
| BET | Brunauer - Emmett - Teller (Method for Surface area Measurement) |
| Btu | British Thermal Unit |
| Cat/CB | Catalyzed Carbon Black |
| DBP | Dibutyl Phthalate |
| GPF | General Purpose Furnace Carbon Black |
| IRB #3 | Industrial Reference Black #3 |
| ISAF-LS | Intermediate Super Abrasion Furnace - Low Structure Carbon Black |
| MT | Medium Thermal Carbon Black |
| MVI | Measuring System for Haake Viscometer |
| n.d. | Not determined |
| nm | Nanometer |
| NV | Measuring system for Haake Viscometer |
| rpm | Revolutions per minute |
| SEM | Scanning Electron Microscope |
| SL-90 | A grade of Semi-reinforcing Furnace (SRF) Carbon Black produced only by Ashland Chemical Company |
| SRF | Semi-Reinforcing Furnace Carbon Black |
| Statex MT | A Medium Thermal Carbon Black produced by the Columbian Chemicals Division of Cities Service Company |
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# SECTION I

## INTRODUCTION

Missile systems constitute a significant portion of the defensive capabilities of the United States. A major constraint on the range and deliverability of new existing missile systems is the size of the air frame.

Recognition of this problem has led to the development of new technology in the area of high energy fuels for missiles. Fuels developed under this program are characterized by having at least 140,000 Btu/gallon and some select fuels have in excess of 160,000 Btu/gallon. A continuing developmental effort has translated this fuels technology into an expanded operational envelope. The value of high density fuels is dependent on the increased volumetric heat content being directly translatable into increased range. For missions of a certain range, the cost of total missile system redesign is saved by the use of high energy fuels.

The state of the art in liquid fuels technology has provided a JP-10 fuel of 141,000 Btu/gallon at a pricing structure that is cost effective for use in current missile systems. Fuels based on hydrogenated norbornadiene dimer (RJ-5), having 161,000 Btu/gallon heat content, however, are not entirely cost effective due to the high cost of norbornadiene and the expense in converting the norbornadiene to RJ-5.

The problem facing the fuels industry and the Armed Forces is to develop a fuel having 180,000 Btu/gallon (or greater) heat content and to demonstrate that the cost of production of the newly developed fuel is compatible with the increased performance of the missile systems on a cost-benefit basis.

Carbon slurry fuels offer one approach to achieving a high heat content fuel. A slurry of 65 weight percent medium thermal (MT) carbon black has a net heat content of about 182,000 Btu/gallon. However, a viable carbon slurry fuel must show high combustion efficiency under operational conditions and recent combustion tests of carbon slurries containing MT carbon black have resulted in poor combustion efficiencies. The reduction of the carbon black particle size, or the incorporation of a combustion catalyst might improve the combustion efficiency.

There are many factors that must be considered in the development of an optimized carbon slurry fuel. The objective of this five-month

program was to initiate the evaluation of two factors which could significantly influence combustion efficiency.

1) The impact of carbon particle size on combustion.

2) The impact on combustion of a catalyst dispersed in the carbon black.

Ashland's role in achieving the above objective has been to conduct the following activities:

1) Preparation and characterization of six carbon slurry fuels containing carbon blacks representing a broad particle size range.

2) Preparation and characterization of two carbon slurry fuels with a combustion catalyst incorporated in the carbon black.

These fuels have been prepared for the purpose of conducting comparative combustion tests to determine the effects of carbon particle size and combustion catalysis. Proprietary technology belonging to the Ashland Chemical Company has been used for the production of catalyzed carbon blacks and for the preparation of carbon slurries. The combustion tests were to be done by the AiResearch Manufacturing Company of Arizona under a separate Air Force contract.

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## SECTION II

## TECHNICAL PROGRAM SUMMARY

This five-month carbon slurry fuel development program was directed toward two objectives: 1) the evaluation of the impact of a catalyst dispersed in the carbon black on combustion of a slurry, and 2) the evaluation of the impact of carbon particle size on combustion. The role of Ashland Chemical Company was to prepare and characterize carbon slurry fuels to be used in achieving the above objectives. The combustion tests were to be done by AiResearch Manufacturing Company of Arizona under a separate contract.

Catalyzed carbon blacks and a control sample were prepared for the purpose of evaluating the effectiveness of the catalyst in the improvement of carbon slurry combustion. Two batches (100 pounds each) of carbon black containing 0.21 and 0.99 percent iron catalyst highly dispersed within the carbon structure were produced in one of Ashland's carbon black research reactors. A carbon black without iron catalyst was also prepared in the same manner, to serve as a base line in the combustion tests. Proprietary technology belonging to Ashland Chemical Company was used to produce the catalyzed carbon blacks. Each carbon black was thoroughly characterized by the standard methods used in the carbon black industry, including examination by Transmission Electron Microscopy (TEM). Slurries (five gallons each) were prepared using state-of-the-art dispersing technology at a carbon black loading of 25 weight percent, with JP-10 as the liquid carrier.

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A total of nine carbon slurries (5 gallons each) were prepared and characterized. Three gallons of each slurry were sent to J.R. McCoy at Wright-Patterson Air Force Base for physical evaluation. Two gallons of each slurry were sent to T. W. Bruce at AiResearch Manufacturing Company of Arizona for combustion testing.

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## TECHNICAL DISCUSSION

### A. BACKGROUND

#### 1. Production of Carbon Black

Carbon black is formed by the incomplete combustion of many organic substances, either solid, liquid, or gaseous. The generic term "carbon black" refers to a group of industrial products consisting of carbonaceous materials of fine particle size made by procedures denoted as furnace, channel, thermal, and lamp. Carbon black materials are composed essentially of elemental carbon, and exist as spherical particles of colloidal sizes that are coalesced into aggregates of higher structure.

Furnace blacks are by far the largest group of commercially available materials, and are prepared by partial combustion of heavy hydrocarbon liquids. Channel blacks are manufactured by impingement of natural gas flames on cold channel irons. Thermal blacks are produced by thermal decomposition of natural gas, while acetylene black, a special type of thermal black, is made by exothermic decomposition of acetylene. Lamp black is made by burning hydrocarbons in open shallow pans.

The furnace process produces blacks with a wide range of particle diameters from 20-100 nanometers. This wider range affords some 30 different standard grades of furnace black which are used in a variety of applications. Furnace blacks generally exist as aggregates of fused spherical particles.

Thermal blacks consist mostly of single spherical particles with diameters of 150-400 nanometers. The structure (level of aggregation) is virtually non-existent.

The physical properties of the carbon black, especially particle size, have a major impact on the properties of the carbon slurry. In a given series of carbon blacks, as the particle size is reduced, the viscosity of the slurries will dramatically increase. Also, at higher loadings non-Newtonian rheological behavior may be observed.

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in a typical engine combustor is a critical factor. One objective of this program is to compare the combustion behavior of slurries containing carbon blacks representing a wide range of particle sizes. This aspect will be discussed later in this report.

Another approach to improve the combustion efficiency of the carbon is to add a combustion catalyst to the carbon slurry fuel. Attempts at Exxon to improve the combustion of a 30% Statex MT slurry with homogeneous catalyst (1000 ppm; Mn, Fe, Pb, Zr) showed no significant improvement (Reference 1). Slurries prepared by Suntech, Inc. containing a homogeneous catalyst were reported to show some qualitative improvement in combustion behavior (Reference 2). These results leave some doubts as to the effectiveness of a homogeneous catalyst. Exxon also found that two heterogeneous catalysts (1% Pt or 1% Pd impregnated on Statex MT carbon black) did not significantly enhance carbon burnout (Reference 3).

One objective of this program is to evaluate the combustion of slurries that have a catalyst intimately incorporated in the carbon black. With this method, catalyst will always be available at the surface of the burning particle. The carbon burn-out time should be substantially reduced if the mechanism is kinetically controlled. Several candidates have been considered for the catalyst in this initial study. Such catalysts effectively reduce smoke in turbine engine exhausts (Reference 4). Carbon (and especially coal) gasification is catalyzed by a variety of catalysts (Reference 5). A carbon black containing a dispersed catalyst should have improved combustion efficiency.

The catalyst-loaded carbon blacks for this research and development program were produced with a reduced-scale version of a commercial furnace reactor. The oil feedstock is atomized into a combustion chamber that is preheated with a natural gas flame. The oil decomposes and forms carbon black which passes through a 6-8 ft. tunnel before being quenched with water. The product is further cooled and collected in bag filters. The method for incorporating catalysts with the carbon black remains proprietary technology of Ashland Chemical Company.

In order to produce the special carbon blacks for this program, it was necessary to reassemble the carbon black research reactor located in the pilot plant facility. Several major components of the system were installed and some minor repiping was completed. The first catalyzed carbon blacks were produced in June 1980.

## 2. Preparation of Carbon Slurries

Carbon blacks differ largely because of their various particle sizes, and the degree of aggregation or structure. The effect of

in a typical engine combustor is a critical factor. One objective of this program is to compare the combustion behavior of slurries containing carbon blacks representing a wide range of particle sizes. This aspect will be discussed later in this report.

Another approach to improve the combustion efficiency of the carbon is to add a combustion catalyst to the carbon slurry fuel. Attempts at Exxon to improve the combustion of a 30% Statex MT slurry with homogeneous catalyst (1000 ppm; Mn, Fe, Pb, Zr) showed no significant improvement (Reference 1). Slurries prepared by Suntech, Inc. containing a homogeneous catalyst were reported to show some qualitative improvement in combustion behavior (Reference 2). These results leave some doubts as to the effectiveness of a homogeneous catalyst. Exxon also found that two heterogeneous catalysts (1% Pt or 1% Pd impregnated on Statex MT carbon black) did not significantly enhance carbon burnout (Reference 3).

One objective of this program is to evaluate the combustion of slurries that have a catalyst intimately incorporated in the carbon black. With this method, catalyst will always be available at the surface of the burning particle. The carbon burn-out time should be substantially reduced if the mechanism is kinetically controlled. Several candidates have been considered for the catalyst in this initial study. Such catalysts effectively reduce smoke in turbine engine exhausts (Reference 4). Carbon (and especially coal) gasification is catalyzed by a variety of catalysts (Reference 5). A carbon black containing a dispersed catalyst should have improved combustion efficiency.

The catalyst-loaded carbon blacks for this research and development program were produced with a reduced-scale version of a commercial furnace reactor. The oil feedstock is atomized into a combustion chamber that is preheated with a natural gas flame. The oil decomposes and forms carbon black which passes through a 6-8 ft. tunnel before being quenched with water. The product is further cooled and collected in bag filters. The method for incorporating catalysts with the carbon black remains proprietary technology of Ashland Chemical Company.

In order to produce the special carbon blacks for this program, it was necessary to reassemble the carbon black research reactor located in the pilot plant facility. Several major components of the system were installed and some minor repiping was completed. The first catalyzed carbon blacks were produced in June 1980.

## 2. Preparation of Carbon Slurries

Carbon blacks differ largely because of their various particle sizes, and the degree of aggregation or structure. The effect of

these two properties along with the surface area, density, percent moisture content, all affect the properties of the final carbon black products. The smaller diameter channel blacks, for example, are used in high quality inks and natural rubber truck tire treads. The relatively large thermal blacks, on the other hand, can be loaded into rubber in large quantities for reducing unit cost since carbon black costs less than rubber. Since furnace blacks can be made in many particle sizes with varying structures, they lend themselves to almost all rubber products, such as tire treads, caracass and mechanical goods as well as inks, plastics and other applications.

The particle diameter is the single most important property and is used to define the various grades of carbon black. The particle diameter can be measured using electron microscopy techniques, or can be calculated from the tint and absorbtometer dibutyl phthalate absorption values.

The carbon black structure or degree of aggregation of small spherical primary units is measured by DBP oil absorption. High absorption numbers indicate greater structure and aggregation of the carbon black microspheres. In slurries of carbon black greater structure usually results in a higher viscosity.

In the combustion of carbon slurry fuels, the question of combustion efficiency versus carbon black particle size is yet unanswered. Experimental and theoretical evidence on the combustion time of carbon particles suggests that the rate of combustion of particles less than 10 microns (10,000 nanometers) in diameter is controlled by surface kinetics, while the rate of combustion of particles greater than 10 microns in diameter is controlled by oxygen diffusion (Reference 6). Carbon black particles, which have primary particle diameters less than 1 micron, should have combustion times less than 5 milliseconds. It has recently been suggested that during the combustion of carbon slurries, the carbon black particles coalesce into agglomerates of 10 to 100 microns in diameter (Reference 7). Poor combustion efficiency and lack of catalytic enhancement of carbon burnout were attributed to the formation of such large carbon black agglomerates. The effect of carbon black particle size on carbon slurry combustion still needs additional investigation.

One of the objectives of this program was to prepare several carbon slurry fuels with a wide range of carbon particle sizes (i.e. 30-300 nm) while minimizing the effects of structure and surface area. Four commercially available carbon blacks have

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One of the objectives of this program was to prepare several carbon slurry fuels with a wide range of carbon particle sizes (i.e. 30-300 nm) while minimizing the effects of structure and surface area. Four commercially available carbon blacks have

been selected. The key physical properties are given below. The characterization methods are described in Appendix A.

Carbon Black Properties

| Grade | ASTM Code | Calc. Avg. Particle Dia. nm | ASTM Tint (% of IRB#3) | DBP Absorp. $cm^3/g$ | ASTM Iodine Number mg/g |
|---|---|---|---|---|---|
| ISAF-LS | N219 | 30 | 117 | 0.78 | 115 |
| GPF | N660 | 56 | 60 | 0.91 | 36 |
| SRF | (SL-90) | 103 | 38 | 0.58 | 24 |
| MT | N990 | 300 | 24 | 0.36 | 10 |

For the comparison of the combustion properties of the four different carbon blacks, it is most desirable to have all the slurries at the same carbon loading in JP-10. The heat content goal for carbon slurry fuels is 180,000 Btu/gallon, which requires a carbon loading of 60-65 wt.%. With the current technology, it is nearly impossible to produce a fluid slurry at that loading with the small particle size carbon blacks such as N219. Therefore, for the purpose of comparison, one slurry of each of the four carbon blacks was prepared at a carbon loading of 25 wt.%. This represents the approximate maximum loading of N219 that would afford a fluid, workable carbon slurry fuel. In addition, two highly loaded slurries of SL-90 (60 wt.%) and N990 (65 wt.%) have been provided for combustion evaluation.

Research at the Ashland Chemical Company has demonstrated that stable, highly dispersed carbon slurry fuels with reasonable viscosities can be prepared. In order to meet the time constraints of this program, existing proprietary technology has been used for the preparation of the carbon slurry fuels to be provided for combustion testing. The process consists of dispersing the carbon black in JP-10 with the aid of a surfactant (to be designated as surfactant AA), followed by thorough milling to afford a highly dispersed stable carbon slurry.

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III.

B. Production and Characterization of Catalyzed Carbon Black

1. Carbon Black Production

The catalyzed carbon blacks were produced with a reduced scale version of a commercial furnace reactor. The oil feedstock is atomized into a combustion chamber that is preheated by a natural gas flame. The oil decomposes and forms a carbon black which passes through a 6-8 foot tunnel before being quenched with water. The product is further cooled and collected in bag filters. The method for incorporating catalysts with the carbon black remains proprietary technology.

A number of 30 to 100 pound batches of iron catalyzed carbon black were prepared. Two batches of catalyzed carbon black with the desired iron concentration and a control batch of carbon black (made in the same reactor but without catalyst) were selected for combustion testing as carbon slurries. The carbon blacks and their respective slurries are identified below.

| Carbon Black Reference No. | Catalyst | Slurry Reference No. |
|---|---|---|
| CBPP-5 | none | 3908-21 |
| CBPP-12 | 0.21% Fe | 3908-22 |
| CBPP-10 | 0.99% Fe | 3908-9 |

2. Carbon Black Characterization

The catalyzed carbon blacks were characterized by the tests most commonly used in the carbon black industry. The tests are described in Appendix A and include the following:

a. Iodine Adsorption Number
b. Dibutyl Phthalate Absorption Number
c. Tint Strength
d. Extractables by Toluene Discoloration
e. Heating Loss

In addition, the surface area was determined by BET nitrogen adsorption; and the particle size distribution was determined by Transmission Electron Microscopy.

Table 1 summarizes the characterization data for the three carbon blacks prepared in the research reactor and the typical carbon black properties for the four commercially available carbon blacks

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TABLE 1

Carbon Black Properties

| Carbon Black Type | N990 (Statex MT) | SL-90 | N660 | N219 | CBPP-5 (Control) | CBPP-12 | CBPP-10 |
|---|---|---|---|---|---|---|---|
| Catalyst Content (wt. %) | none | none | none | none | 0.02 | 0.21 | 0.99 |
| ASTM Iodine No. (mg/g) | 10 | 24 | 36 | 115 | 94 | 113 | 87 |
| B.E.T. $N_2$ Surface Area ($m^2/g$) | n.d. | n.d. | n.d. | n.d. | 133 | 145 | 119 |
| DBP Absorption ($cm^3/g$) | 0.36 | 0.58 | 0.91 | 0.78 | 0.57 | 0.54 | 1.24 |
| ASTM Tint (% of IRB #3) | 24 | 38 | 60 | 117 | 122 | 127 | 105 |
| Calc. Avg. Particle Diameter (nm) | 300 | 103 | 56 | 30 | 37 | 37 | 26 |
| Arithmetic Mean Diameter by TEM (nm) | n.d. | n.d. | n.d. | n.d. | 24 | 21 | 27 |
| Percent Heat Loss | 0.5 | 0.2 | 0.4 | 0.7 | 0.8 | 0.01 | 0.02 |
| Percent Transmittance (Toluene Extraction) | 99 | 90 | 95 | 97 | 3 | 6 | 2 |

n.d. = not determined

of different particle size. The three prepared carbon blacks have relatively high surface areas (Iodine number and nitrogen absorption) and small particle sizes. These materials are similar to the commercial N219 grade. However, two samples (CBPP-5 and -12) have low structure, as indicated by the DBP absorption values of 0.57 and 0.54 $cm^3/g$ respectively. Sample CBPP-10 has a high structure as indicated by the DBP absorption of 1.24 $cm^3/g$.

The low value for Percent Transmittance on Toluene Extraction indicate that the prepared carbon blacks have substantially more oil on the surface than the commercial carbon blacks. This is a characteristic of the reduced dimensions of the research reactor. The actual increase in the amount of oil on the carbon black is quite small and should have very little effect on the carbon slurry properties. The low values for the Percent Heat Loss test indicate that the samples do not contain excess adsorbed water.

Samples of the two catalyzed carbon blacks were examined by high resolution Transmission Electron Microscopy (TEM). Micrographs are shown in Figures 1 to 5. No unusual features are observed. The carbon black does not have numerous detectable catalyst particles on it, as one would see if it were impregnated with catalyst.

Samples of the respective carbon slurries were used for the determination of the particle size distribution by TEM, and bar graphs are given in Figures 6 to 8. There are discrepancies between the Arithmetic Mean Diameter by TEM and the Calculated Average Particle Diameter (see Table 1). The latter value is calculated from the DBP Absorption and the ASTM Tint number. Techniques used in the production of the carbon blacks may have altered the surface properties, which could possibly affect the tint or the DBP measurements. The result would be an inaccurate value for the Calculated Particle Diameter.

An ideal catalyzed carbon black would have a particle size of about 100 nm (i.e. SL-90). This should afford the optimum viscosity at high carbon loadings (50-60 weight percent). Unfortunately the carbon black research reactor is only capable of producing carbon blacks with small average particle size (20 to 40 nm) such as N219, N220, N326, etc. If the catalyzed carbon blacks produced in this reactor system show enhanced combustion properties, the process could be scaled-up to a commercial reactor. Catalyst could then be incorporated into a SL-90 type carbon, which would be produced in large batches.

Figure 1 CBPP-12, 0.21 Cat/CB
TEM 100,000 x
1 cm = 100 nm

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TEM 100,000 x
1 cm = 100 nm

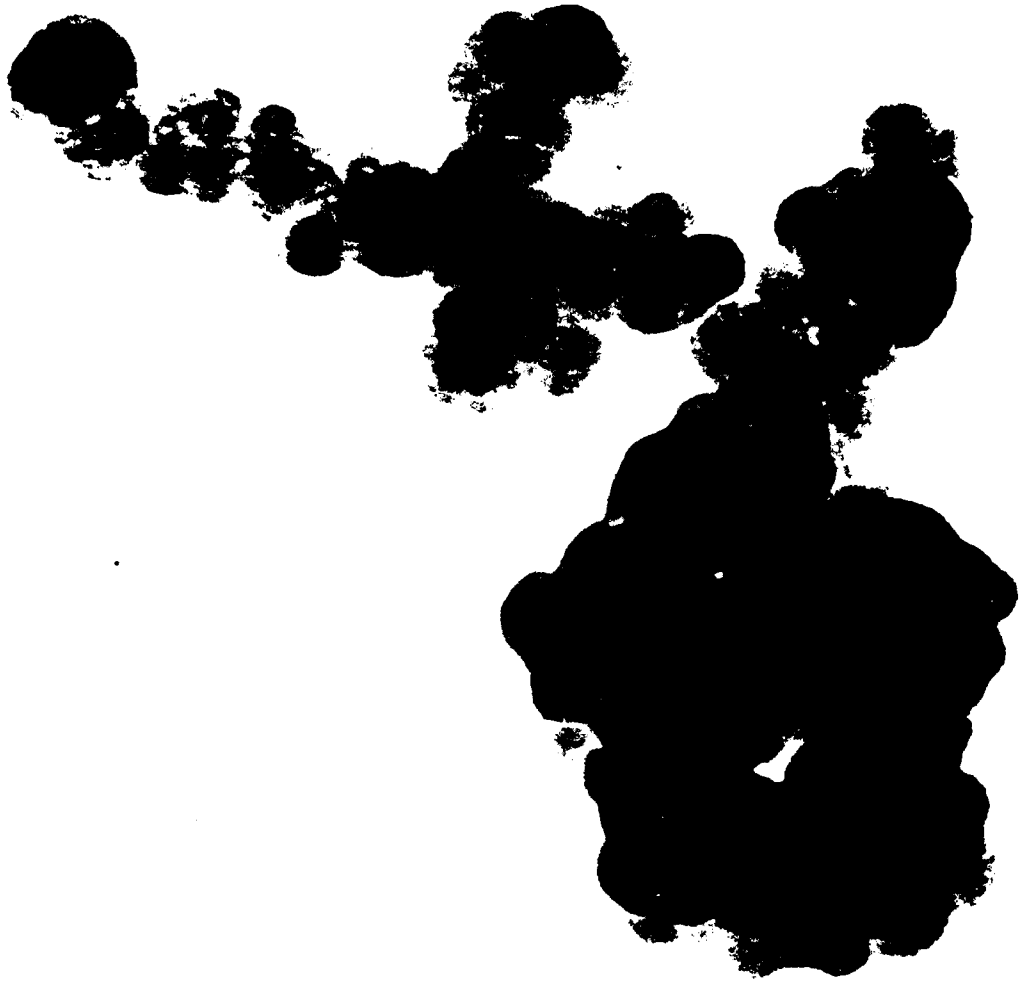
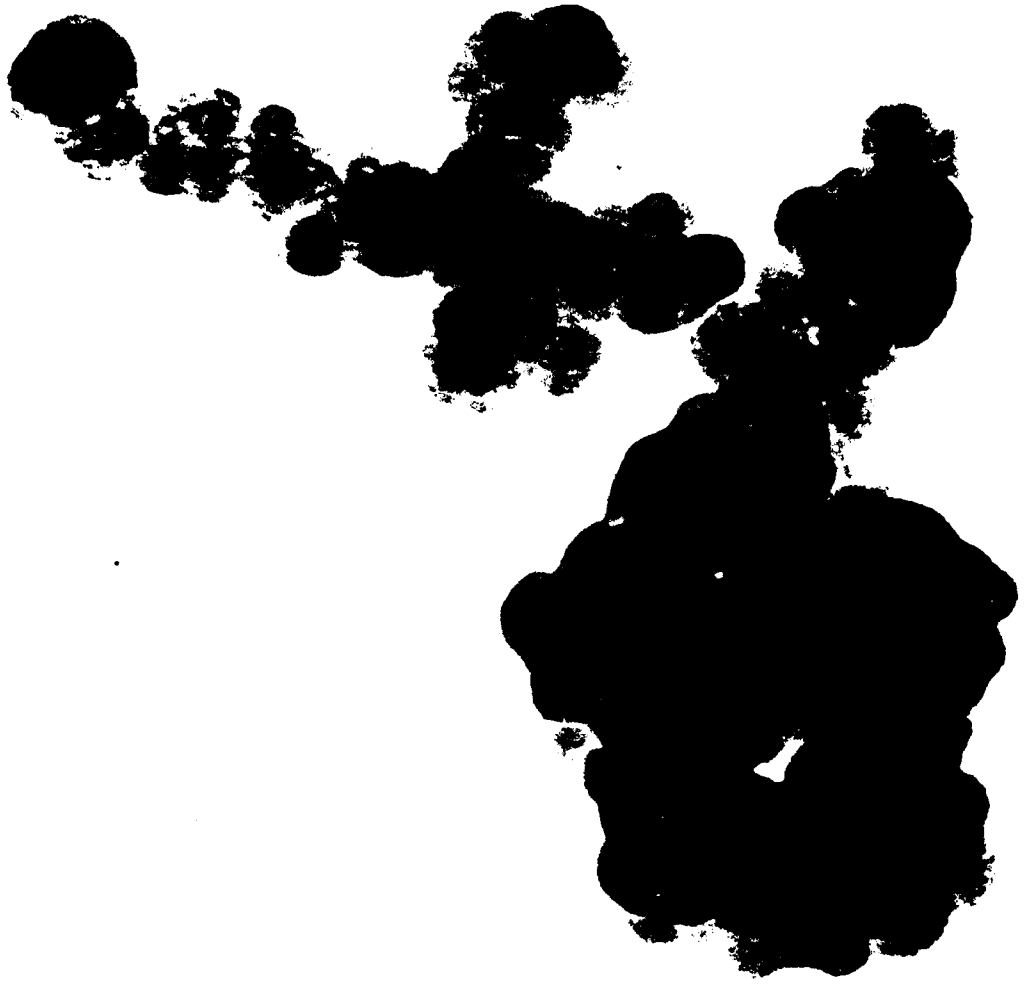


Figure 2. CBPP-12, 0.21% Cat/CB
TEM 310,000 x
1 cm = 32.2 nm



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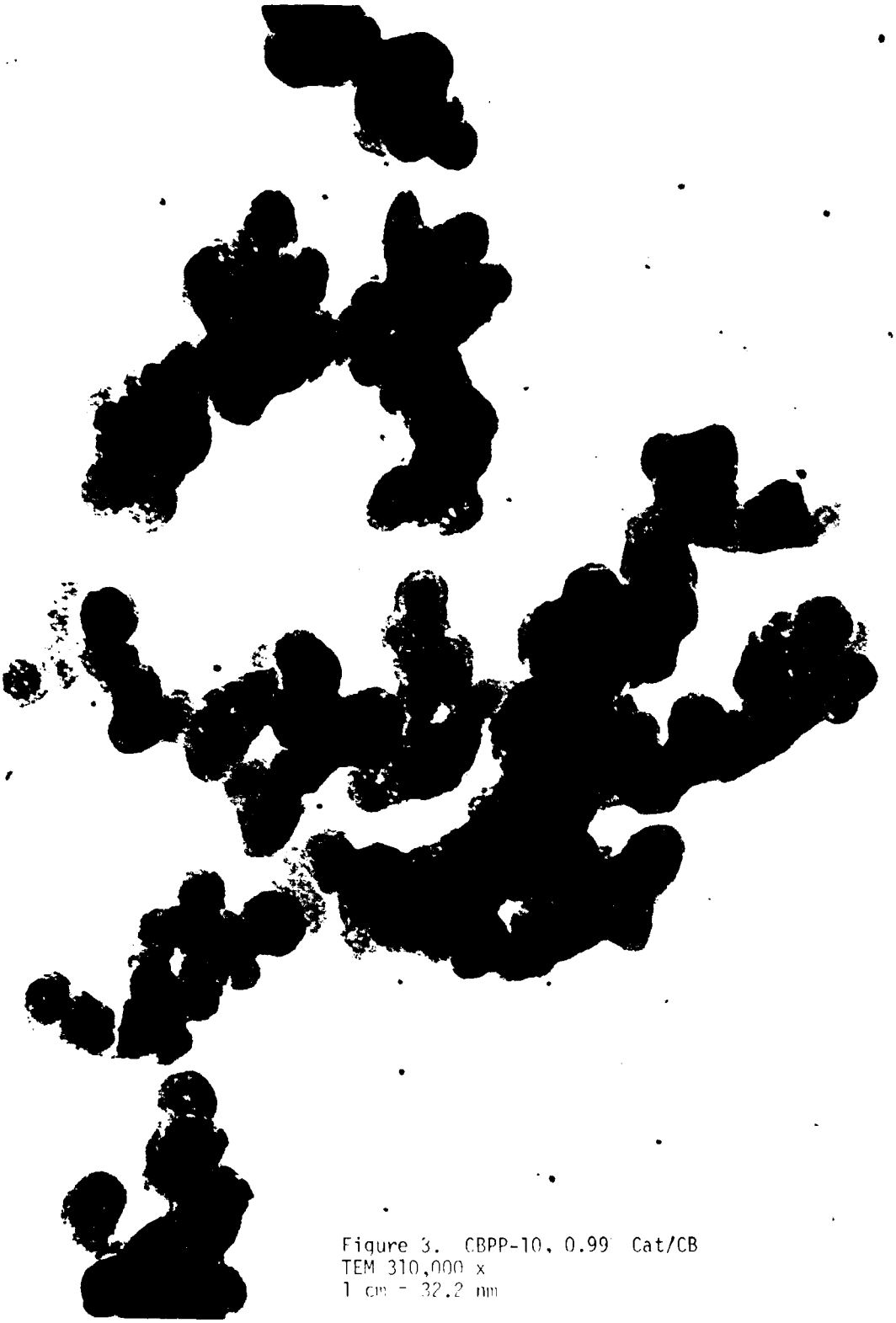
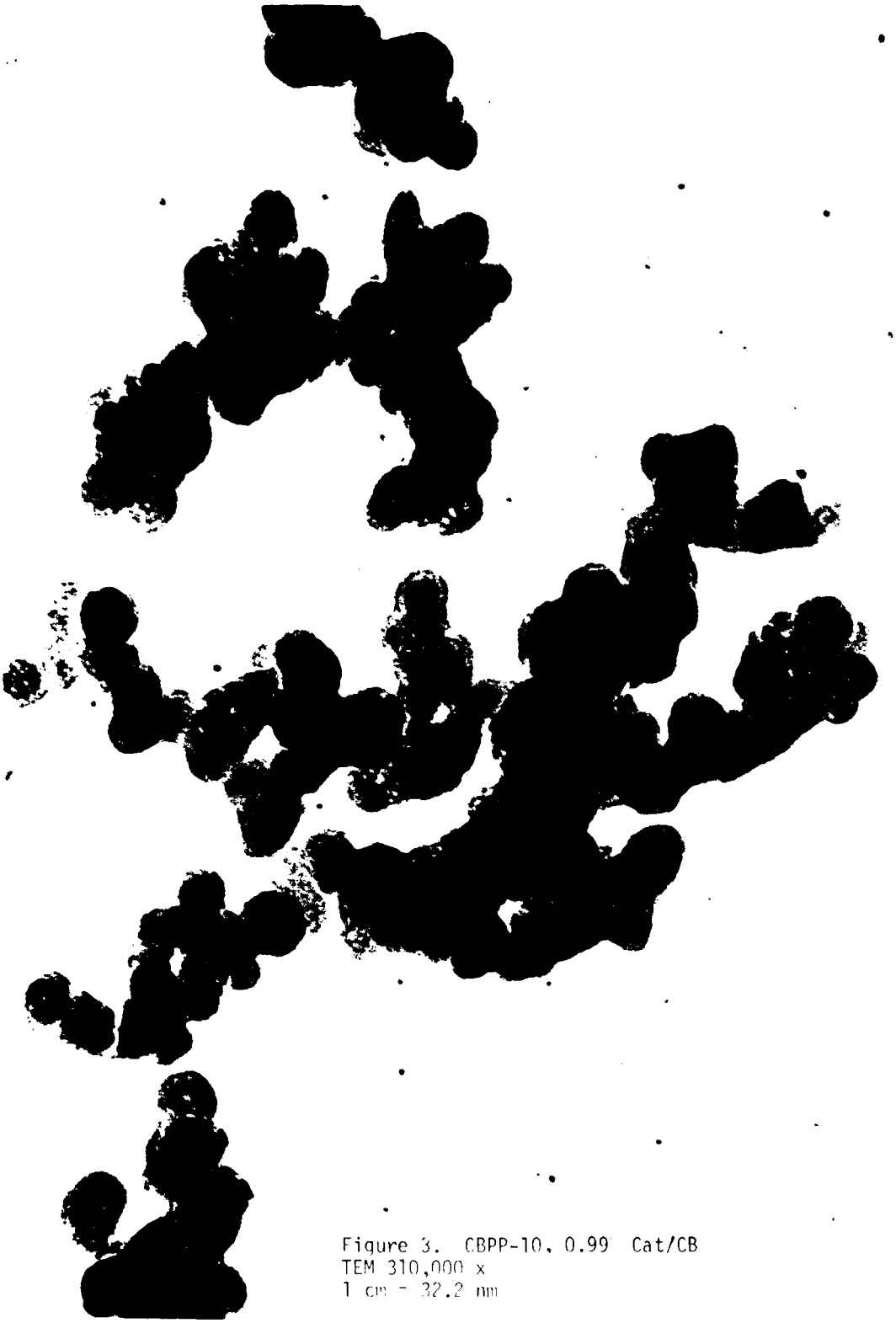


Figure 3. CBPP-10, 0.99 Cat/CB
TEM 310,000 x
1 cm = 32.2 nm



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TEM 310,000 x
1 cm = 32.2 nm

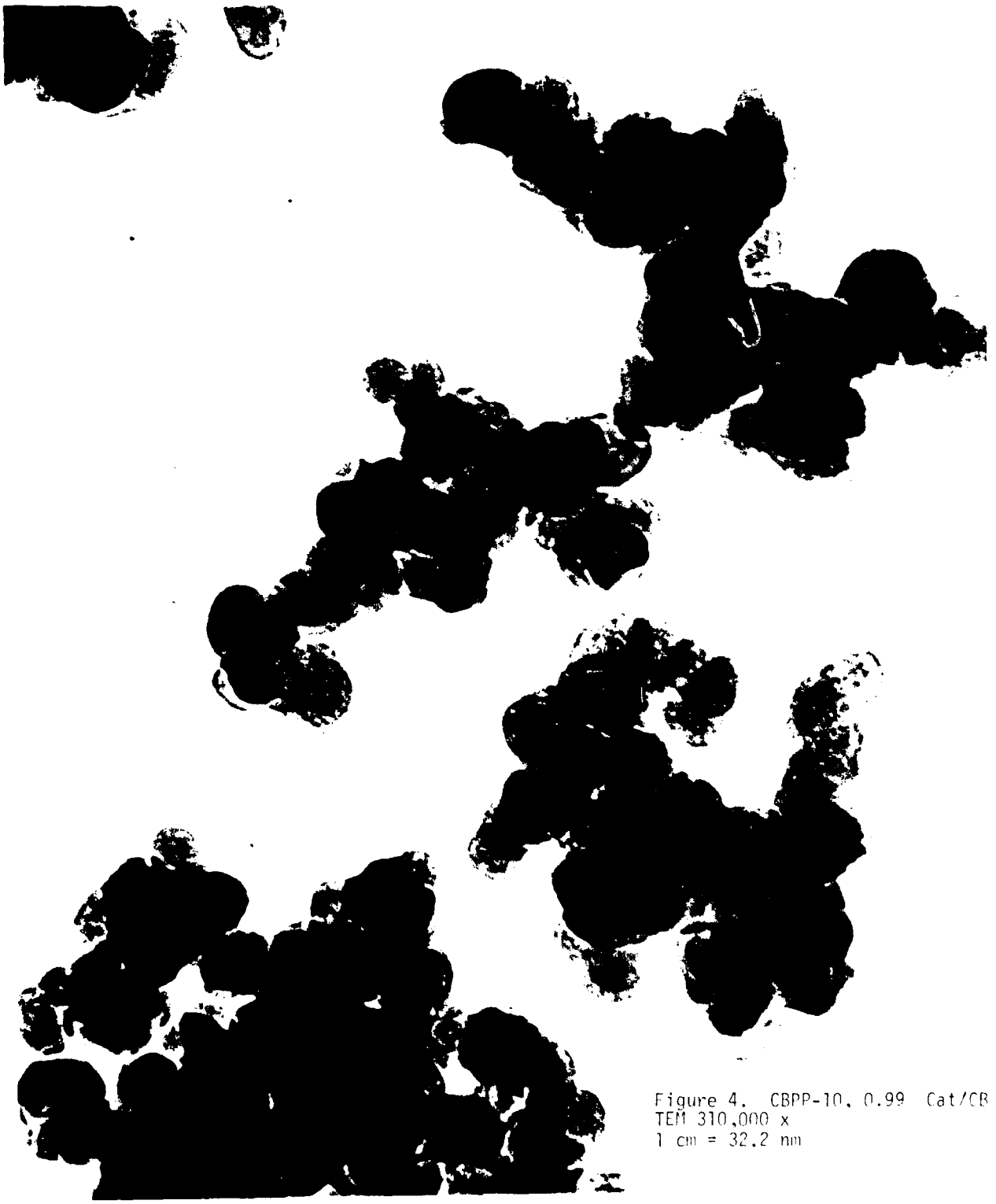

Figure 4. CBPP-10, 0.99 Cat/CB
TEM 310,000 x
1 cm = 32.2 nm

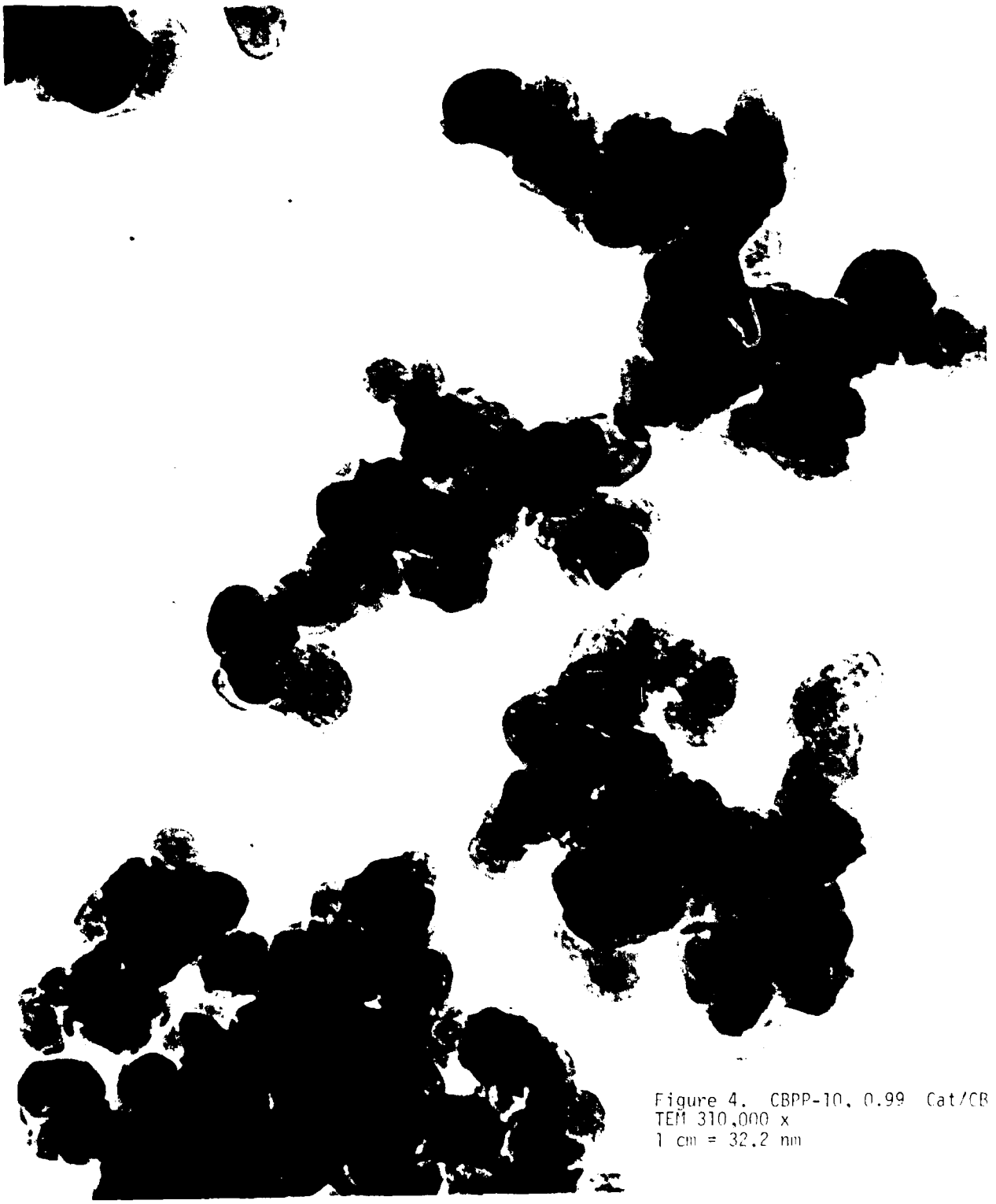

Figure 4. CBPP-10, 0.99 Cat/CB
TEM 310,000 x
1 cm = 32.2 nm

Figure 5. CBPP-10, 0.99% Cat/CB
TEM 310,000 x
1 cm = 32.2 nm

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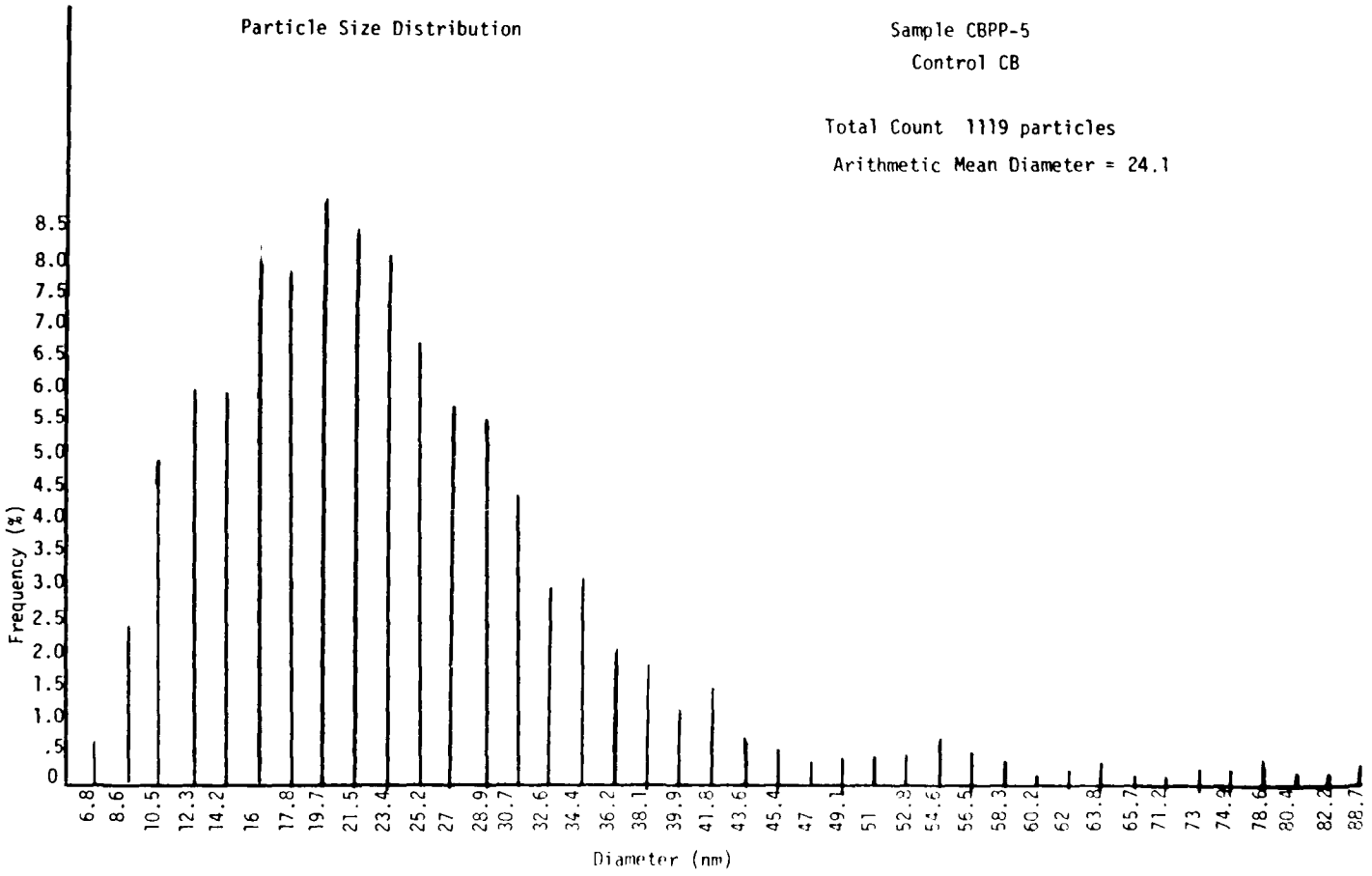
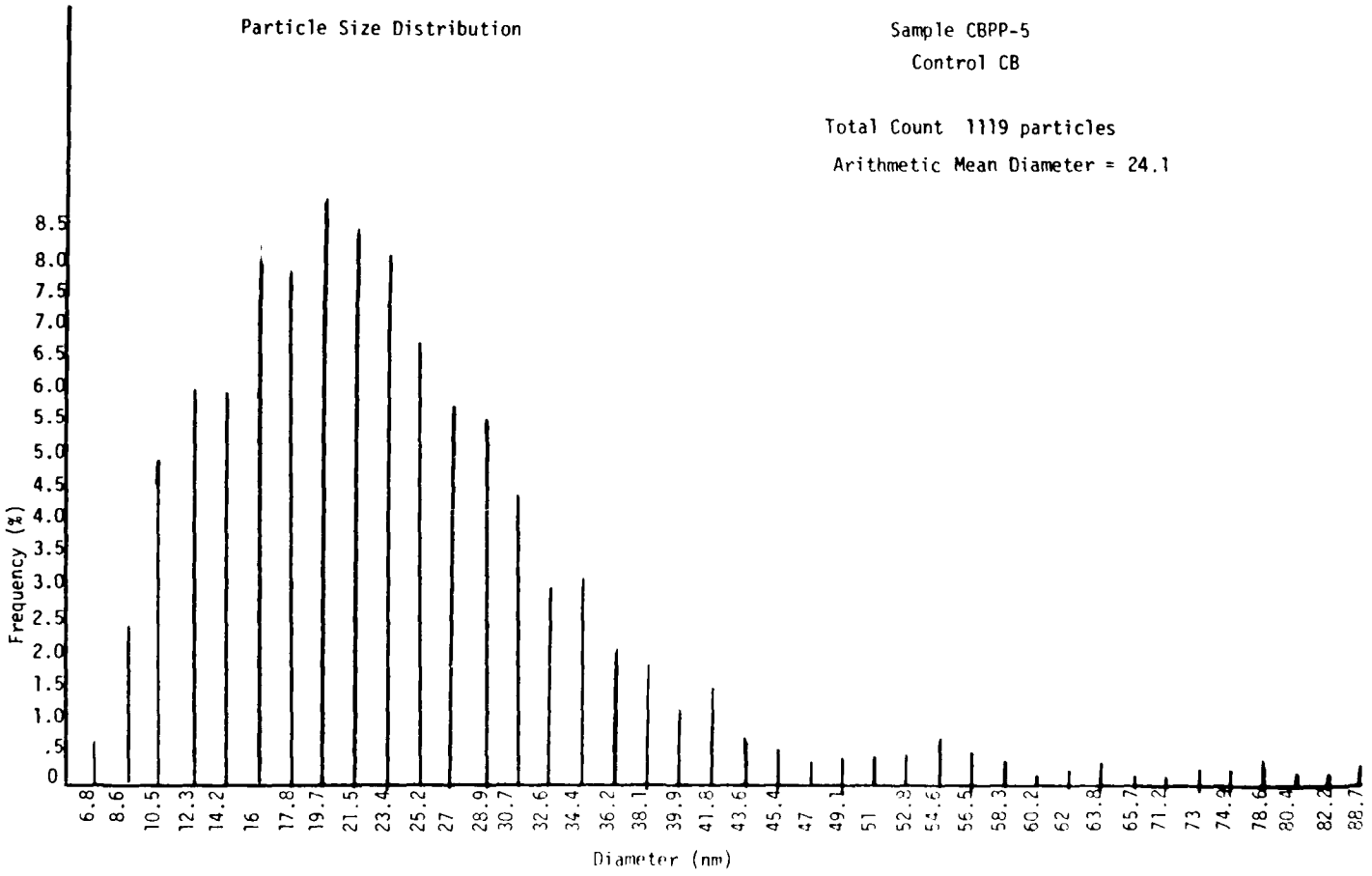



Figure 6. Carbon Black Particle Size Distribution by TEM

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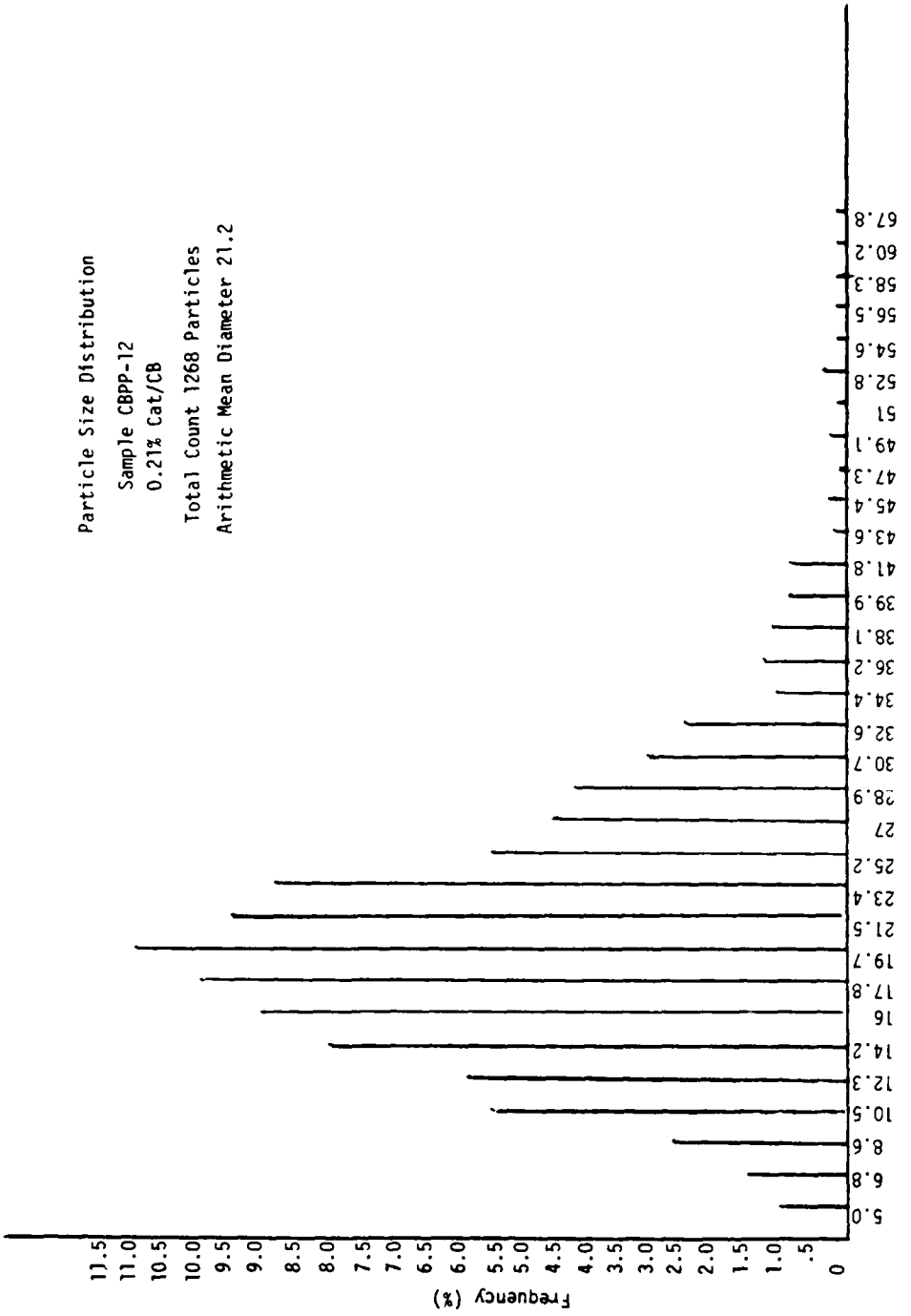


Figure 7. Carbon Black Particle Size Distribution by TEM

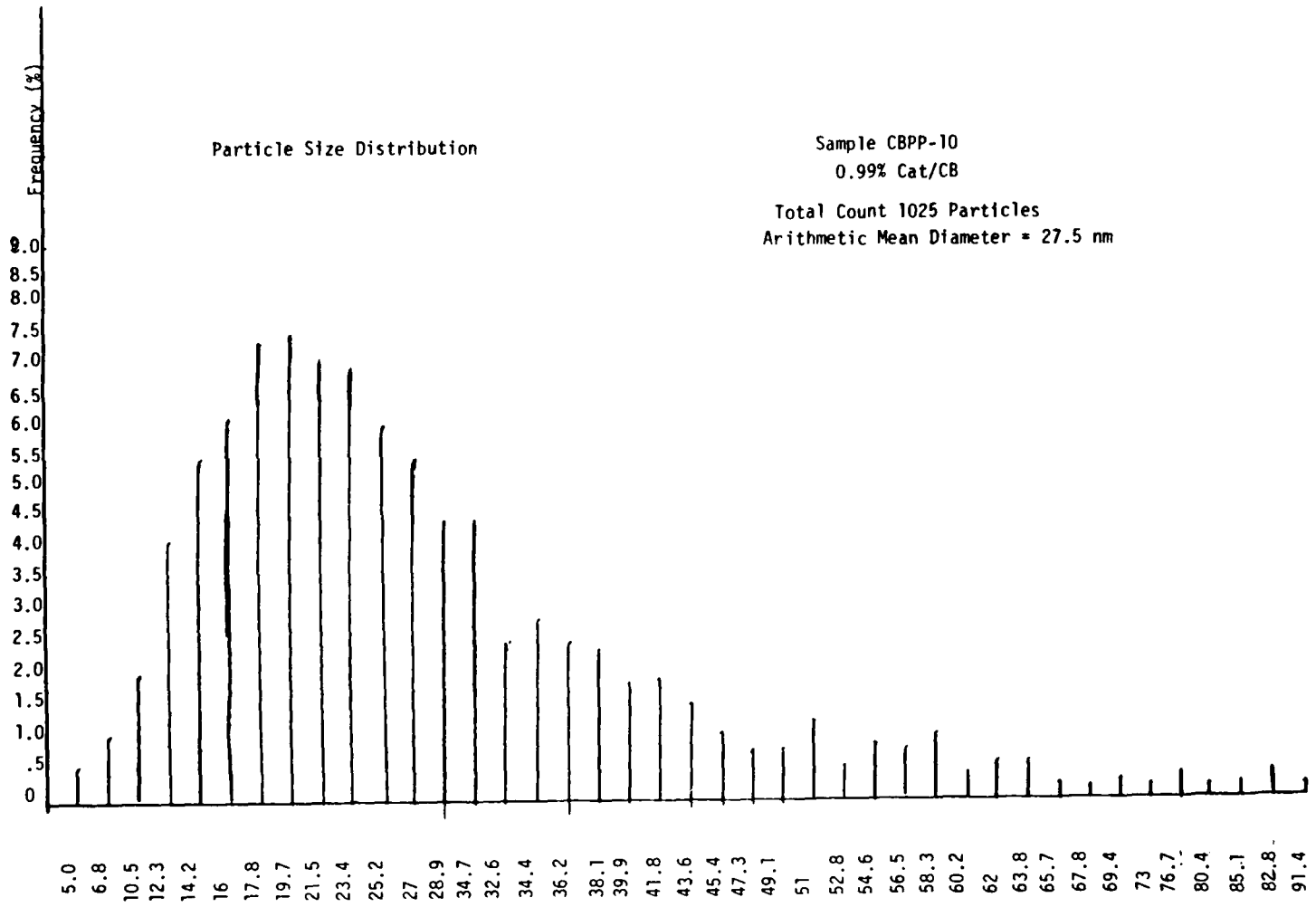


Figure 8. Carbon Black Particle Size Distribution by TEM

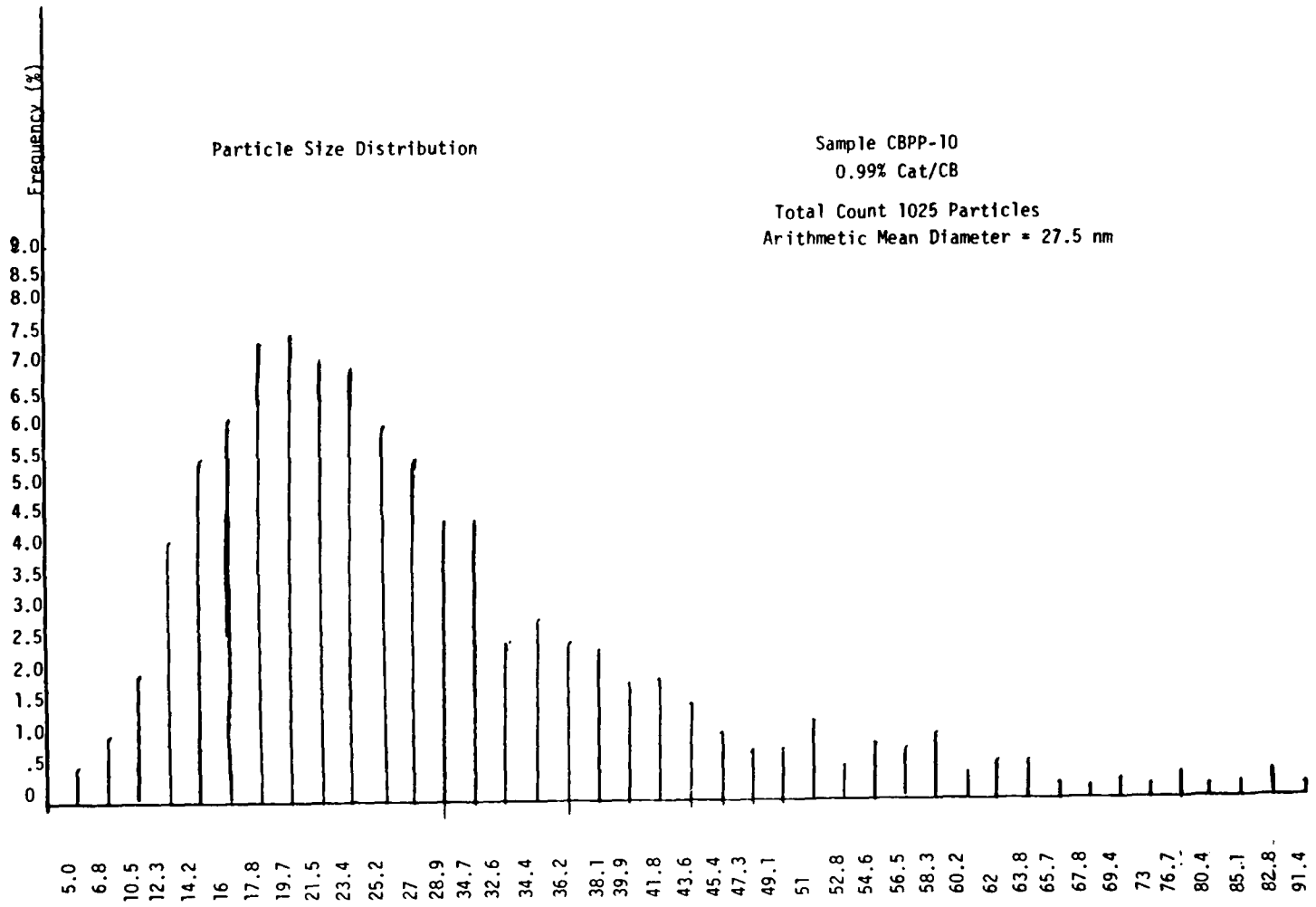


Figure 8. Carbon Black Particle Size Distribution by TEM

III.

## C. Preparation and Characterization of Carbon Slurries

### 1. Carbon Slurry Preparation

For the purpose of comparing the combustion properties, a slurry of each type of carbon black was prepared at a loading of 25 weight percent. In addition, a slurry of 65 weight percent Statex MT and a slurry of 60 weight percent SL-90 were prepared. Surfactant AA was used at a concentration of 5 weight percent relative to the total slurry weight for the commercial carbon blacks, and 6 weight percent for the experimental carbon blacks. These surfactant concentrations were about the level required to afford the optimum viscosity, except for Statex MT and SL-90 which required less surfactant.

Each carbon slurry was prepared by the standard procedure in which the total weighed amount of dispersant was dissolved in the JP-10; while mixing with a high speed disperser, the carbon black was slowly added; the pre-mixed slurry was then ball milled a number of hours. The resulting slurries had a smooth consistency and were relatively stable. No sedimentation or syneresis was observed after centrifugation at 900g for 30 minutes. However, after 2 hours of centrifugation at 900g, a thin, tacky layer of sediment (1 to 2 mm) was detected in most of the slurries. After storage at ambient conditions for 4 to 6 weeks, a similar thin, tacky layer of sediment (1 to 2 mm) was detected in most of the slurries. The stability of these slurries was not a critical problem, but was the object of some concern. In the short term (several months) stability should not be a problem in the combustion tests.

### 2. Carbon Slurry Properties

The physical and thermochemical properties for each carbon slurry are presented in Table 2. A description of each characterization method is provided in Appendix B. The properties presented in Table 2 include:

a. Composition of the slurry
b. Brookfield viscosity at 25°C (10 $sec^{-1}$ shear rate)
c. Haake viscosity at 1°, 25°, 40°C; (200 $sec^{-1}$ shear rate)
d. Density at 25°C
e. Net heat of combustion
f. Centrifuge rating for stability
g. Flash point

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TABLE 2

Carbon Slurry Properties

| Slurry Reference No. | 3856-41 | 3856-64 | 3856-123 | 3856-83 | 3856-77 | 3856-99 | 3908-21 | 3908-22 | 3908-9 |
|---|---|---|---|---|---|---|---|---|---|
| 1. Composition | | | | | | | | | |
| Type carbon black | N990(MT) | SL-90 | N990(MT) | SL-90 | N660 | N219 | Control | 0.21% Cat/CB | 0.99% Cat/CB |
| Reference Number | - | - | - | - | - | - | CBPP-5 | CBPP-12 | CBPP-10 |
| Avg.particle size (nm) | 300 | 103 | 300 | 103 | 56 | 30 | 37 | 37 | 26 |
| Wt. % carbon black | 65 | 60 | 25 | 25 | 25 | 25 | 25 | 25 | 25 |
| Wt. % surfactant AA | 5 | 5 | 5 | 5 | 5 | 5 | 6 | 6 | 6 |
| Wt. % JP-10 | 30 | 35 | 70 | 70 | 70 | 69 | 69 | 69 | 69 |
| 2. Brookfield Viscosity (cp) 25°C, 100 RPM | | | | | | | | | |
| 10 $sec^{-1}$ shear rate | 326 | 1128 | 41 | 39 | 74 | 580 | 505 | 1017 | 636 |
| 3. Haake Viscosity (cp) | | | | | | | | | |
| 200 $sec^{-1}$ shear rate, 1°C | 390 | - | 7 | 12 | 50 | 142 | 110 | 140 | 120 |
| 25°C | 120 | - | 5 | 11 | 33 | 110 | 92 | 140 | 120 |
| 40°C | 120 | 440 | 5 | 6 | 20 | 106 | 85 | 150 | 100 |
| 4. Density at 25°C (lb/gal) (pycnometer cup) | 11.450 | 11.067 | 8.855 | 8.845 | 8.892 | 8.856 | 8.831 | 8.835 | 8.784 |
| 5. Net Heat of Combustion | | | | | | | | | |
| Btu/lb | 15,900 | 15,900 | 17,330 | 17,240 | 17,270 | 17,120 | 17,658 | 17,672 | 17,050 |
| Btu/gal | 182,050 | 175,950 | 153,460 | 152,500 | 153,560 | 151,610 | 155,940 | 156,130 | 149,770 |
| 6. Centrifuge Rating 30 min. at 900G | passed | passed | passed | passed | passed | passed | passed | passed | passed |
| 7. Flash Point (°F) | 128 | 128 | 128 | 127 | 127 | 128 | 128 | 128 | 128 |

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| Wt. % carbon black | 65 | 60 | 25 | 25 | 25 | 25 | 25 | 25 | 25 |
| Wt. % surfactant AA | 5 | 5 | 5 | 5 | 5 | 5 | 6 | 6 | 6 |
| Wt. % JP-10 | 30 | 35 | 70 | 70 | 70 | 69 | 69 | 69 | 69 |
| 2. Brookfield Viscosity (cp) 25°C, 100 RPM | | | | | | | | | |
| 10 $sec^{-1}$ shear rate | 326 | 1128 | 41 | 39 | 74 | 580 | 505 | 1017 | 636 |
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| 200 $sec^{-1}$ shear rate, 1°C | 390 | - | 7 | 12 | 50 | 142 | 110 | 140 | 120 |
| 25°C | 120 | - | 5 | 11 | 33 | 110 | 92 | 140 | 120 |
| 40°C | 120 | 440 | 5 | 6 | 20 | 106 | 85 | 150 | 100 |
| 4. Density at 25°C (lb/gal) (pycnometer cup) | 11.450 | 11.067 | 8.855 | 8.845 | 8.892 | 8.856 | 8.831 | 8.835 | 8.784 |
| 5. Net Heat of Combustion | | | | | | | | | |
| Btu/lb | 15,900 | 15,900 | 17,330 | 17,240 | 17,270 | 17,120 | 17,658 | 17,672 | 17,050 |
| Btu/gal | 182,050 | 175,950 | 153,460 | 152,500 | 153,560 | 151,610 | 155,940 | 156,130 | 149,770 |
| 6. Centrifuge Rating 30 min. at 900G | passed | passed | passed | passed | passed | passed | passed | passed | passed |
| 7. Flash Point (°F) | 128 | 128 | 128 | 127 | 127 | 128 | 128 | 128 | 128 |

Most of the slurries have a composition of 25 weight percent carbon black, 5 or 6 weight percent Surfactant AA, and JP-10 as the balance. Slurry 3856-41 contains 65 weight percent Statex MT, and slurry 3856-64 contains 60 weight percent SL-90.

The Brookfield Viscometer is used routinely in the laboratory for a quick evaluation of the viscous properties of each slurry. The instrument is best used for comparing one slurry relative to another, since, due to the design of the instrument, neither the shear rates or viscosities are absolute. The shear rate at 100 RPM is estimated to be approximately 10 $sec^{-1}$. Viscosities at 100 RPM are reported in Table 2. The trend is toward higher viscosities for slurries with small particle size carbon black, ard for slurries at a higher carbon loading.

The viscosity at a shear rate of 200 $sec^{-1}$ for each slurry as measured by a Haake RV-1 Rotoviscometer at three temperatures (1°, 25°, and 40°C) is reported in Table 2. The slurries of 25 weight percent of Statex MT and SL-90 have very low viscosities (5-7 cp and 6-12 cp, respectively), nearly approaching the viscosity of neat JP-10. At higher loading (60-65 weight percent), the viscosities are substantially higher. Slurries of smaller particle carbon blacks also have higher viscosities. Slurries 3908-22 and -9 are unique in their behavior in that the viscosity does not progressively increase as the temperature is lowered. Rheology of the carbon slurries will be further discussed below.

The density of each slurry was determined with a pycnometer cup at 25°C and is reported in Table 2. Some small variations in density are observed. Slurry 3908-9 containing 25 weight percent of a carbon black with 0.99 percent catalyst had the lowest density.

Both the gravimetric and volumetric net heat of combustion are reported in Table 2 for each slurry. For the 25 weight percent carbon loaded slurries, the volumetric net heat of combustion ranges from 152,000 to 156,000 Btu/gal, except for 3908-9 which had only 149,770 Btu/gal. This slurry had the lowest density and the lowest gravimetric net heat of combustion.

All slurries passed the stability centrifuge test (30 minutes at 900g), and the flash point of each slurry was at 127-128°F, as shown in Table 2.

C.

3. Carbon Slurry Rheology

The viscosity data obtained with the Brookfield viscometer and the Haake RV-1 Rotoviscometer are tabulated in Table 3. Measurements were made using either a NV or a MVI rotor-beaker assembly with the Haake Rotoviscometer (see Appendix B). At each temperature, the viscosity measurements progressed from the lowest shear rate to the highest and back to the lowest. The rotational speed was manually set at each shear rate and the shear stress reading was taken 30 seconds after the rotor was started. Viscosities are reported in centipoise. Logarithmetic plots of viscosity as a function of shear rate are presented in Figures 9 to 17.

Additional viscosity data was obtained with a Contraves Rheomat 30 rotoviscometer. This instrument is capable of automatically scanning a given shear rate range and plotting the shear stress as a function of the shear rate. Viscosities are then calculated for points along the curve. Figures 9 to 17 are logarithmetic plots of viscosity as a function of shear rate for each of the carbon slurry fuels. The A/A bob and cup measuring system covers a shear rate range of 0 to 662 $sec^{-1}$.

The following observations on the rheology of the carbon slurries are drawn from Table 3 and Figures 9 through 17.

Slurry 3856-41 (65% Statex MT, Figure 9) has moderate viscosity, ranging from 120 to 1000 centipoise, which represents a substantial degree of shear thinning or pseudoplastic behavior. A slight tendency to dilatant behavior occurs at shear rates above 400 $sec^{-1}$.

Slurry 3856-64 (60% SL-90, Figure 10) ranged in viscosity from 700 to 3500 centipoise, which represents a pseudoplastic behavior. The slurry was less thixotropic than one might have expected, considering the high viscosity. Between the shear rates of 100 to 200 centipoise (at 25°C) a sudden dilatant behavior would occur, which caused the shear stress readings to rapidly go off-scale. The exact point at which the dilatant behavior sets in is a function of the temperature and the configuration or gap of the specific measuring system. Of course, the carbon black loading is the critical factor. Subsequent studies have found that the sudden dilatant behavior does not occur between 0 and 600 $sec^{-1}$ shear rate for a 57 weight percent SL-90 slurry as measured by the A/A cup and bob system at 25°C. The sudden dilatant behavior could be a serious problem for pumping and atomizing the carbon slurry fuel. None of the other carbon slurries in this program have behaved in this manner.

C.

3. Carbon Slurry Rheology

The viscosity data obtained with the Brookfield viscometer and the Haake RV-1 Rotoviscometer are tabulated in Table 3. Measurements were made using either a NV or a MVI rotor-beaker assembly with the Haake Rotoviscometer (see Appendix B). At each temperature, the viscosity measurements progressed from the lowest shear rate to the highest and back to the lowest. The rotational speed was manually set at each shear rate and the shear stress reading was taken 30 seconds after the rotor was started. Viscosities are reported in centipoise. Logarithmetic plots of viscosity as a function of shear rate are presented in Figures 9 to 17.

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The following observations on the rheology of the carbon slurries are drawn from Table 3 and Figures 9 through 17.

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Slurry 3856-64 (60% SL-90, Figure 10) ranged in viscosity from 700 to 3500 centipoise, which represents a pseudoplastic behavior. The slurry was less thixotropic than one might have expected, considering the high viscosity. Between the shear rates of 100 to 200 centipoise (at 25°C) a sudden dilatant behavior would occur, which caused the shear stress readings to rapidly go off-scale. The exact point at which the dilatant behavior sets in is a function of the temperature and the configuration or gap of the specific measuring system. Of course, the carbon black loading is the critical factor. Subsequent studies have found that the sudden dilatant behavior does not occur between 0 and 600 $sec^{-1}$ shear rate for a 57 weight percent SL-90 slurry as measured by the A/A cup and bob system at 25°C. The sudden dilatant behavior could be a serious problem for pumping and atomizing the carbon slurry fuel. None of the other carbon slurries in this program have behaved in this manner.

Slurries 3856-123 (25% Statex MT, Figure 11), 3856-83 (25% SL-90, Figure 12) and 3856-77 (25% N660, Figure 13) all had low viscosities and thus had little tendency toward pseudoplastic or thixotropic behavior.

Slurry 3856-99 (25% N219, Figure 14) had high viscosity at low shear rates but showed a substantial shear thinning. The viscosity was slow to rebuild when the shear rate was reduced, thus showing its thixotropic behavior. As one might expect, the viscosity is reduced as the temperature is raised.

Slurries 3908-21 (25% uncatalyzed carbon black, Figure 15), 3908-22 (25% of 0.2% catalyzed carbon black, Figure 16), and 3908-9 (25% of 0.99% catalyzed carbon black, Figure 17) cover a wide range of viscosities due to their pseudoplastic behavior. The slurries were quite thixotropic. These slurries also had an interesting temperature effect since at 40°C the viscosity was higher than at 25°C in the lower shear rate range (9-150 $sec^{-1}$). That trend was reversed above 150 $sec^{-1}$ shear rate and the viscosities were lower at 40°C.

In summary, all of the slurries, except the 60 weight percent SL-90, had viscosities below 160 centipoise at a shear rate of 200 $sec^{-1}$ at 25°C, suggesting that the viscosity of these slurries should not cause problems in pumping or atomizing at temperatures near 25°C. The highly loaded slurries and the slurries containing small particle size carbon blacks tended to have pseudoplastic and thixotropic behavior, which makes them appear to have a thicker, gel-like consistency when not under shear.

Slurries 3856-123 (25% Statex MT, Figure 11), 3856-83 (25% SL-90, Figure 12) and 3856-77 (25% N660, Figure 13) all had low viscosities and thus had little tendency toward pseudoplastic or thixotropic behavior.

Slurry 3856-99 (25% N219, Figure 14) had high viscosity at low shear rates but showed a substantial shear thinning. The viscosity was slow to rebuild when the shear rate was reduced, thus showing its thixotropic behavior. As one might expect, the viscosity is reduced as the temperature is raised.

Slurries 3908-21 (25% uncatalyzed carbon black, Figure 15), 3908-22 (25% of 0.2% catalyzed carbon black, Figure 16), and 3908-9 (25% of 0.99% catalyzed carbon black, Figure 17) cover a wide range of viscosities due to their pseudoplastic behavior. The slurries were quite thixotropic. These slurries also had an interesting temperature effect since at 40°C the viscosity was higher than at 25°C in the lower shear rate range (9-150 $sec^{-1}$). That trend was reversed above 150 $sec^{-1}$ shear rate and the viscosities were lower at 40°C.

In summary, all of the slurries, except the 60 weight percent SL-90, had viscosities below 160 centipoise at a shear rate of 200 $sec^{-1}$ at 25°C, suggesting that the viscosity of these slurries should not cause problems in pumping or atomizing at temperatures near 25°C. The highly loaded slurries and the slurries containing small particle size carbon blacks tended to have pseudoplastic and thixotropic behavior, which makes them appear to have a thicker, gel-like consistency when not under shear.

TABLE 3

Viscosity of Carbon Slurry Fuels

| Slurry Reference | 3856-41 | | | 3856-64 | | | 3856-123 | | |
|---|---|---|---|---|---|---|---|---|---|
| Type of Carbon Black | N990 (MT) | | | SL-90 | | | N990 (MT) | | |
| Weight % Carbon Black | 65 | | | 60 | | | 25 | | |
| **Brookfield Viscosity** | **Temp** | **rpm** | **Viscosity** | **Temp** | **rpm** | **Viscosity** | **Temp** | **rpm** | **Viscosity** |
| in centipoise (cp) at 25°C | 25 | 20 | 464 | 25 | 20 | 1702 | 25 | 20 | 20 |
| 100 rpm = shear rate | 25 | 50 | 387 | 25 | 50 | 1320 | 25 | 50 | 28 |
| at 10 $sec^{-1}$ | 25 | 100 | 326 | 25 | 100 | 1128 | 25 | 100 | 41 |
| **Haake RV-1** | **Temp** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp** | **Shear Rate** | **Viscosity** |
| Viscosity in centipoise (cp) | 40.0 | 174 | 126 | 40.1 | 39 | 554 | 40.0 | 1370 | 5.9 |
| Temperature in °C | 40.0 | 349 | 116 | 40.1 | 58 | 501 | | | |
| Shear rate in $sec^{-1}$ | 40.0 | 523 | 115 | 40.1 | 116 | 449 | | | |
| | 40.0 | 1047 | 119 | 40.1 | 174 | 442 | | | |
| | 40.0 | * 523 | 103 | 40.1 | 349 | O.S. | | | |
| | 40.0 | * 349 | 104 | 40.1 | * 174 | 441 | | | |
| | 40.0 | * 174 | 109 | 40.1 | * 116 | 441 | | | |
| | | | | | * 39 | 520 | | | |
| | 25.0 | 457 | 116 | 27.7 | 39 | 730 | 26.2 | 685 | 5.0 |
| | 25.0 | 685 | 121 | 27.7 | 58 | 678 | 26.2 | 1370 | 6.3 |
| * Rerun | 25.0 | 1370 | 141 | 27.7 | 116 | O.S. | 26.2 | * 685 | 4.7 |
| | 25.0 | * 685 | 108 | 27.7 | * 58 | 670 | | | |
| O.S. = off scale | 25.0 | * 457 | 103 | 27.7 | * 39 | 713 | | | |
| | 1.5 | 152 | 375 | 1.1 | 25 | 1512 | 0.4 | 457 | 7.3 |
| | 1.5 | 228 | 402 | 1.1 | 51 | O.S. | 0.4 | 685 | 7.5 |
| | 1.5 | 457 | O.S. | 1.1 | 25 | 1427 | 0.4 | 1370 | 10.1 |
| | 1.5 | * 152 | 296 | | | | 0.4 | * 685 | 8.8 |
| | | | | | | | 0.4 | * 457 | 8.1 |

TABLE 3

Viscosity of Carbon Slurry Fuels

| Slurry Reference | 3856-41 | | | 3856-64 | | | 3856-123 | | |
|---|---|---|---|---|---|---|---|---|---|
| Type of Carbon Black | N990 (MT) | | | SL-90 | | | N990 (MT) | | |
| Weight % Carbon Black | 65 | | | 60 | | | 25 | | |
| **Brookfield Viscosity** | **Temp** | **rpm** | **Viscosity** | **Temp** | **rpm** | **Viscosity** | **Temp** | **rpm** | **Viscosity** |
| in centipoise (cp) at 25°C | 25 | 20 | 464 | 25 | 20 | 1702 | 25 | 20 | 20 |
| 100 rpm = shear rate | 25 | 50 | 387 | 25 | 50 | 1320 | 25 | 50 | 28 |
| at 10 $sec^{-1}$ | 25 | 100 | 326 | 25 | 100 | 1128 | 25 | 100 | 41 |
| **Haake RV-1** | **Temp** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp** | **Shear Rate** | **Viscosity** |
| Viscosity in centipoise (cp) | 40.0 | 174 | 126 | 40.1 | 39 | 554 | 40.0 | 1370 | 5.9 |
| Temperature in °C | 40.0 | 349 | 116 | 40.1 | 58 | 501 | | | |
| Shear rate in $sec^{-1}$ | 40.0 | 523 | 115 | 40.1 | 116 | 449 | | | |
| | 40.0 | 1047 | 119 | 40.1 | 174 | 442 | | | |
| | 40.0 | * 523 | 103 | 40.1 | 349 | O.S. | | | |
| | 40.0 | * 349 | 104 | 40.1 | * 174 | 441 | | | |
| | 40.0 | * 174 | 109 | 40.1 | * 116 | 441 | | | |
| | | | | | * 39 | 520 | | | |
| | 25.0 | 457 | 116 | 27.7 | 39 | 730 | 26.2 | 685 | 5.0 |
| | 25.0 | 685 | 121 | 27.7 | 58 | 678 | 26.2 | 1370 | 6.3 |
| * Rerun | 25.0 | 1370 | 141 | 27.7 | 116 | O.S. | 26.2 | * 685 | 4.7 |
| | 25.0 | * 685 | 108 | 27.7 | * 58 | 670 | | | |
| O.S. = off scale | 25.0 | * 457 | 103 | 27.7 | * 39 | 713 | | | |
| | 1.5 | 152 | 375 | 1.1 | 25 | 1512 | 0.4 | 457 | 7.3 |
| | 1.5 | 228 | 402 | 1.1 | 51 | O.S. | 0.4 | 685 | 7.5 |
| | 1.5 | 457 | O.S. | 1.1 | 25 | 1427 | 0.4 | 1370 | 10.1 |
| | 1.5 | * 152 | 296 | | | | 0.4 | * 685 | 8.8 |
| | | | | | | | 0.4 | * 457 | 8.1 |

TABLE 3 (Continued)

VISCOSITY OF CARBON SLURRY FUELS

| Slurry Reference No. | 3856-83 | | | 3856-77 | | | 3856-99 | | |
|---|---|---|---|---|---|---|---|---|---|
| Type of Carbon Black | SL-90 | | | N 660 | | | N 219 | | |
| Wt.% Carbon Black | 25 | | | 25 | | | 25 | | |
| **Brookfield Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** |
| in centipoise (cp) at 25°C | 25 | 20 | 24 | 25 | 20 | 50 | 25 | 20 | 940 |
| 100 rpm = shear rate of | 25 | 50 | 28 | 25 | 50 | 56 | 25 | 50 | 622 |
| 10 $sec^{-1}$ | 25 | 100 | 39 | 25 | 100 | 74 | 25 | 100 | 580 |
| **Haake RV-1** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** |
| Viscosity in centipoise (cp) | 40.0 | 349 | 6.1 | 40.2 | 116 | 20.6 | 40.2 | 116 | 159 |
| Temperature in °C | 40.0 | 523 | 6.3 | 40.2 | 174 | 20.9 | 40.2 | 174 | 118 |
| Shear rate in $sec^{-1}$ | 40.0 | 1047 | 6.2 | 40.2 | 349 | 20.3 | 40.2 | 349 | 76 |
| | 40.0 | 1570 | 6.1 | 40.2 | 523 | 19.6 | 40.2 | 523 | 58 |
| | 40.0 | *1047 | 6.4 | 40.2 | *523 | 19.5 | 40.2 | 1047 | 40 |
| | 40.0 | * 523 | 6.5 | 40.2 | *349 | 19.9 | 40.2 | 1570 | 32 |
| | 40.0 | * 349 | 6.1 | 40.2 | *174 | 20.1 | 40.2 | 3140 | 24 |
| | | | | 40.2 | *116 | 20.9 | 40.2 | *1047 | 33 |
| * Rerun | | | | | | | 40.2 | * 523 | 41 |
| | | | | | | | 40.2 | * 349 | 53 |
| | 25.8 | 349 | 10.4 | 25.8 | 58 | 39.5 | 25.7 | 174 | 120 |
| | 25.8 | 523 | 9.9 | 25.8 | 116 | 36.1 | 25.7 | 349 | 76 |
| | 25.8 | 1047 | 9.0 | 25.8 | 174 | 33.9 | 25.7 | 523 | 61 |
| | 25.8 | 1570 | 8.7 | 25.8 | 349 | 30.9 | 25.7 | 1047 | 45 |
| | 25.8 | *1047 | 9.1 | 25.8 | 523 | 29.7 | 25.7 | 1570 | 37 |
| | 25.8 | * 523 | 10.2 | 25.8 | * 349 | 30.2 | 25.7 | 3140 | 28 |
| | 25.8 | * 349 | 10.4 | 25.8 | * 174 | 31.9 | 25.7 | *1047 | 32 |
| | | | | 25.8 | * 116 | 34.1 | 25.7 | * 523 | 38 |
| | | | | 25.8 | * 58 | 35.5 | | | |
| | 1.0 | 174 | 12.4 | 0.0 | 58 | 49.5 | 0.8 | 116 | 215 |
| | 1.0 | 349 | 13.6 | 0.0 | 116 | 51.5 | 0.8 | 174 | 158 |
| | 1.0 | 523 | 13.9 | 0.0 | 174 | 50.1 | 0.8 | 349 | 105 |
| | 1.0 | 1047 | 13.7 | 0.0 | * 174 | 49.6 | 0.8 | 523 | 85 |
| | 1.0 | *1047 | 13.6 | 0.0 | * 116 | 49.4 | 0.8 | 1047 | 63 |
| | 1.0 | * 523 | 13.4 | 0.0 | * 58 | 46.4 | 0.8 | 1570 | 52 |
| | 1.0 | * 349 | 12.8 | | | | 0.8 | 3140 | 38 |
| | 1.0 | * 174 | 11.4 | | | | 0.8 | *1047 | 42 |
| | | | | | | | 0.8 | * 523 | 47 |
| | | | | | | | 0.8 | * 349 | 53 |

TABLE 3 (Continued)

VISCOSITY OF CARBON SLURRY FUELS

| Slurry Reference No. | 3856-83 | | | 3856-77 | | | 3856-99 | | |
|---|---|---|---|---|---|---|---|---|---|
| Type of Carbon Black | SL-90 | | | N 660 | | | N 219 | | |
| Wt.% Carbon Black | 25 | | | 25 | | | 25 | | |
| **Brookfield Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** |
| in centipoise (cp) at 25°C | 25 | 20 | 24 | 25 | 20 | 50 | 25 | 20 | 940 |
| 100 rpm = shear rate of | 25 | 50 | 28 | 25 | 50 | 56 | 25 | 50 | 622 |
| 10 $sec^{-1}$ | 25 | 100 | 39 | 25 | 100 | 74 | 25 | 100 | 580 |
| **Haake RV-1** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** |
| Viscosity in centipoise (cp) | 40.0 | 349 | 6.1 | 40.2 | 116 | 20.6 | 40.2 | 116 | 159 |
| Temperature in °C | 40.0 | 523 | 6.3 | 40.2 | 174 | 20.9 | 40.2 | 174 | 118 |
| Shear rate in $sec^{-1}$ | 40.0 | 1047 | 6.2 | 40.2 | 349 | 20.3 | 40.2 | 349 | 76 |
| | 40.0 | 1570 | 6.1 | 40.2 | 523 | 19.6 | 40.2 | 523 | 58 |
| | 40.0 | *1047 | 6.4 | 40.2 | *523 | 19.5 | 40.2 | 1047 | 40 |
| | 40.0 | * 523 | 6.5 | 40.2 | *349 | 19.9 | 40.2 | 1570 | 32 |
| | 40.0 | * 349 | 6.1 | 40.2 | *174 | 20.1 | 40.2 | 3140 | 24 |
| | | | | 40.2 | *116 | 20.9 | 40.2 | *1047 | 33 |
| * Rerun | | | | | | | 40.2 | * 523 | 41 |
| | | | | | | | 40.2 | * 349 | 53 |
| | 25.8 | 349 | 10.4 | 25.8 | 58 | 39.5 | 25.7 | 174 | 120 |
| | 25.8 | 523 | 9.9 | 25.8 | 116 | 36.1 | 25.7 | 349 | 76 |
| | 25.8 | 1047 | 9.0 | 25.8 | 174 | 33.9 | 25.7 | 523 | 61 |
| | 25.8 | 1570 | 8.7 | 25.8 | 349 | 30.9 | 25.7 | 1047 | 45 |
| | 25.8 | *1047 | 9.1 | 25.8 | 523 | 29.7 | 25.7 | 1570 | 37 |
| | 25.8 | * 523 | 10.2 | 25.8 | * 349 | 30.2 | 25.7 | 3140 | 28 |
| | 25.8 | * 349 | 10.4 | 25.8 | * 174 | 31.9 | 25.7 | *1047 | 32 |
| | | | | 25.8 | * 116 | 34.1 | 25.7 | * 523 | 38 |
| | | | | 25.8 | * 58 | 35.5 | | | |
| | 1.0 | 174 | 12.4 | 0.0 | 58 | 49.5 | 0.8 | 116 | 215 |
| | 1.0 | 349 | 13.6 | 0.0 | 116 | 51.5 | 0.8 | 174 | 158 |
| | 1.0 | 523 | 13.9 | 0.0 | 174 | 50.1 | 0.8 | 349 | 105 |
| | 1.0 | 1047 | 13.7 | 0.0 | * 174 | 49.6 | 0.8 | 523 | 85 |
| | 1.0 | *1047 | 13.6 | 0.0 | * 116 | 49.4 | 0.8 | 1047 | 63 |
| | 1.0 | * 523 | 13.4 | 0.0 | * 58 | 46.4 | 0.8 | 1570 | 52 |
| | 1.0 | * 349 | 12.8 | | | | 0.8 | 3140 | 38 |
| | 1.0 | * 174 | 11.4 | | | | 0.8 | *1047 | 42 |
| | | | | | | | 0.8 | * 523 | 47 |
| | | | | | | | 0.8 | * 349 | 53 |

TABLE 3 (Continued)

VISCOSITY OF CARBON SLURRY FUELS

| Slurry Reference No. | 3908-21 | | | 3908-22 | | | 3908-9 | | |
|---|---|---|---|---|---|---|---|---|---|
| Type of Carbon Black | Un-Cat C.B. | | | 0.2% Cat. C.B. | | | 1.0% Cat. C.B. | | |
| Weight % Carbon Black | 25 | | | 25 | | | 25 | | |
| **Brookfield Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** | **Temp.** | **rpm** | **Viscosity** |
| in centipoise (cp) at 25°C | 25 | 20 | 978 | 25 | 20 | 2546 | 25 | 20 | 1448 |
| 100 rpm = shear rate | 25 | 50 | 690 | 25 | 50 | 1526 | 25 | 50 | 1052 |
| at 10 $sec^{-1}$ | 25 | 100 | 505 | 25 | 100 | 1017 | 25 | 100 | 636 |
| **Haake RV-1** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** | **Temp.** | **Shear Rate** | **Viscosity** |
| Viscosity in centipoise(cp) | 40.1 | 9 | 624 | 40.1 | 9 | 1967 | 40.1 | 17 | 334 |
| Temperature in °C | 40.1 | 25 | 334 | 40.1 | 25 | 819 | 40.1 | 25 | 270 |
| Shear rate in $sec^{-1}$ | 40.1 | 51 | 219 | 40.1 | 51 | 467 | 40.1 | 51 | 211 |
| | 40.1 | 152 | 99 | 40.1 | 76 | 323 | 40.1 | 76 | 157 |
| | 40.1 | 228 | 73 | 40.1 | 152 | 188 | 40.1 | 152 | 121 |
| | 40.1 | 457 | 50 | 40.1 | *152 | 168 | 40.1 | 228 | 89 |
| | 40.1 | 685 | 39 | 40.1 | * 76 | 253 | 40.1 | *228 | 71 |
| | 40.1 | *457 | 36 | 40.1 | * 51 | 356 | 40.1 | *152 | 70 |
| | 40.1 | *152 | 46 | 40.1 | * 25 | 627 | 40.1 | * 76 | 77 |
| * Rerun | 40.1 | * 51 | 73 | 40.1 | * 9 | 1785 | 40.1 | * 51 | 84 |
| | 26.8 | 25 | 207 | 26.3 | 9 | 1190 | 27.0 | 25 | 220 |
| | 26.8 | 51 | 200 | 26.3 | 25 | 677 | 27.0 | 51 | 209 |
| | 26.8 | 76 | 159 | 26.3 | 51 | 394 | 27.0 | 76 | 174 |
| | 26.8 | 152 | 113 | 26.3 | 76 | 287 | 27.0 | 152 | 145 |
| | 26.8 | 228 | 79 | 26.3 | 152 | 175 | 27.0 | 228 | 112 |
| | 26.8 | 457 | 59 | 26.3 | 228 | 126 | 27.0 | *228 | 89 |
| | 26.8 | 685 | 44 | 26.3 | *152 | 119 | 27.0 | *152 | 87 |
| | 26.8 | *457 | 39 | 26.3 | * 51 | 218 | 27.0 | * 76 | 90 |
| | 26.8 | *228 | 39 | 26.3 | * 25 | 317 | 27.0 | * 51 | 91 |
| | 26.8 | *152 | 42 | 26.3 | * 9 | 766 | | | |
| | 0.9 | 9 | 519 | 0.7 | 9 | 1270 | 0.2 | 9 | 751 |
| | 0.9 | 25 | 375 | 0.7 | 17 | 887 | 0.2 | 17 | 677 |
| | 0.9 | 51 | 285 | 0.7 | 25 | 634 | 0.2 | 25 | 560 |
| | 0.9 | 76 | 202 | 0.7 | 51 | 394 | 0.2 | 51 | 406 |
| | 0.9 | 152 | 139 | 0.7 | 76 | 282 | 0.2 | 76 | 291 |
| | 0.9 | 228 | 98 | 0.7 | * 76 | 231 | 0.2 | * 76 | 223 |
| | 0.9 | *228 | 80 | 0.7 | * 51 | 238 | 0.2 | * 51 | 219 |
| | 0.9 | *152 | 75 | 0.7 | * 25 | 281 | 0.2 | * 25 | 230 |
| | 0.9 | * 76 | 78 | 0.7 | * 17 | 348 | 0.2 | *17 | 243 |
| | 0.9 | * 51 | 81 | 0.7 | * 9 | 425 | | | |

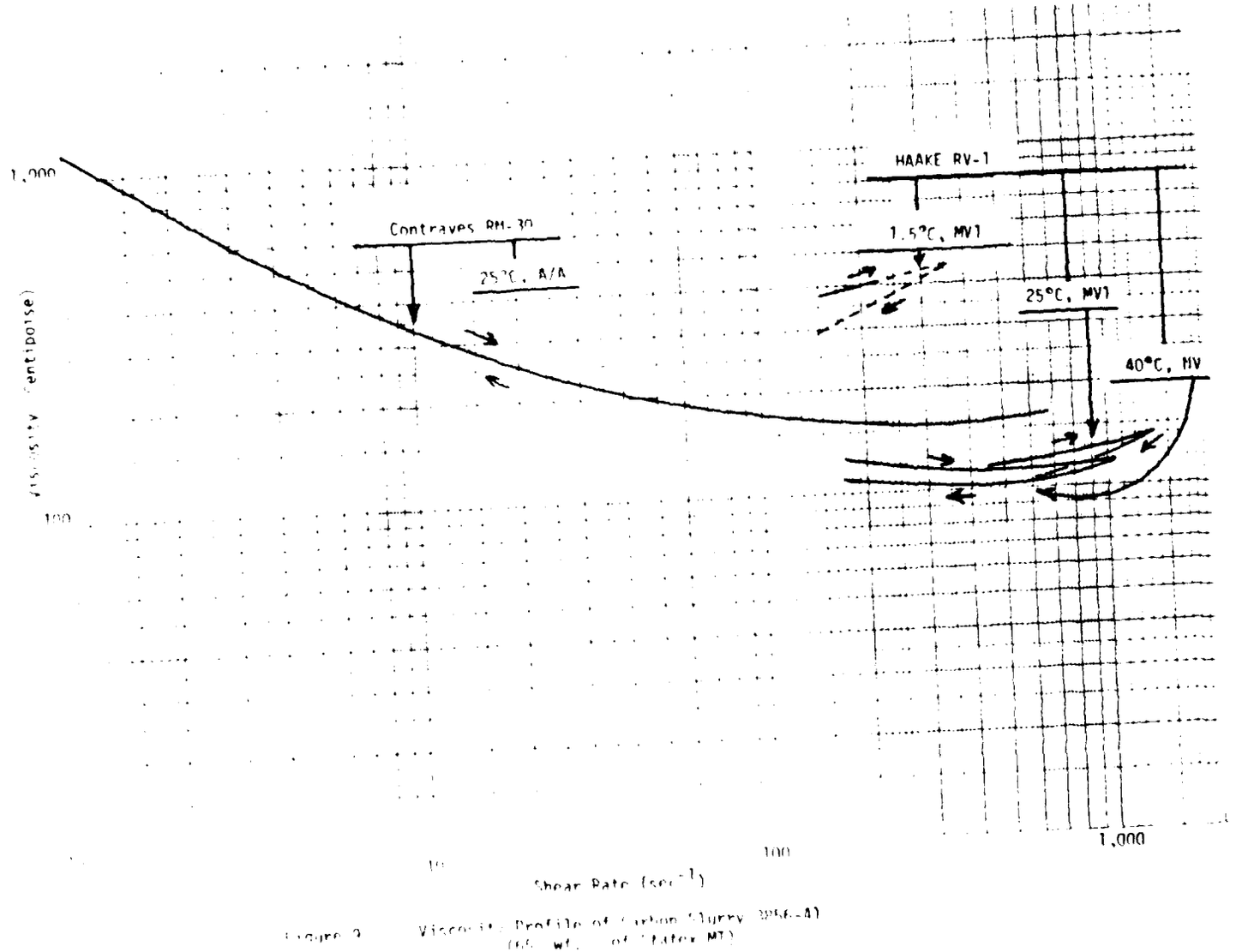
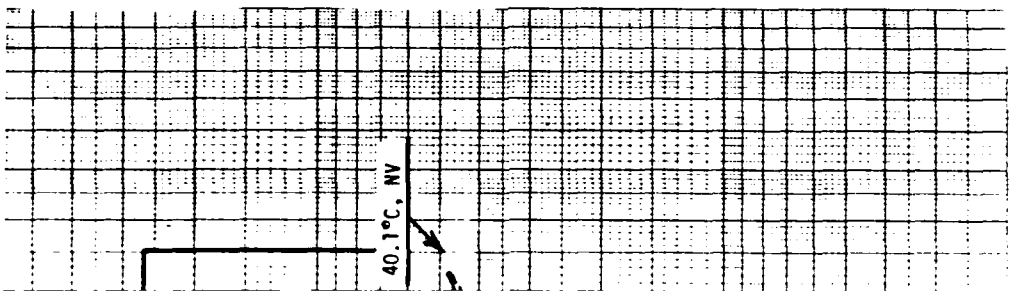



Figure 3 Viscosity Profile of Carbon Slurry (3256-41) (66 wt. % of Pater MT)


40.1°C, NV

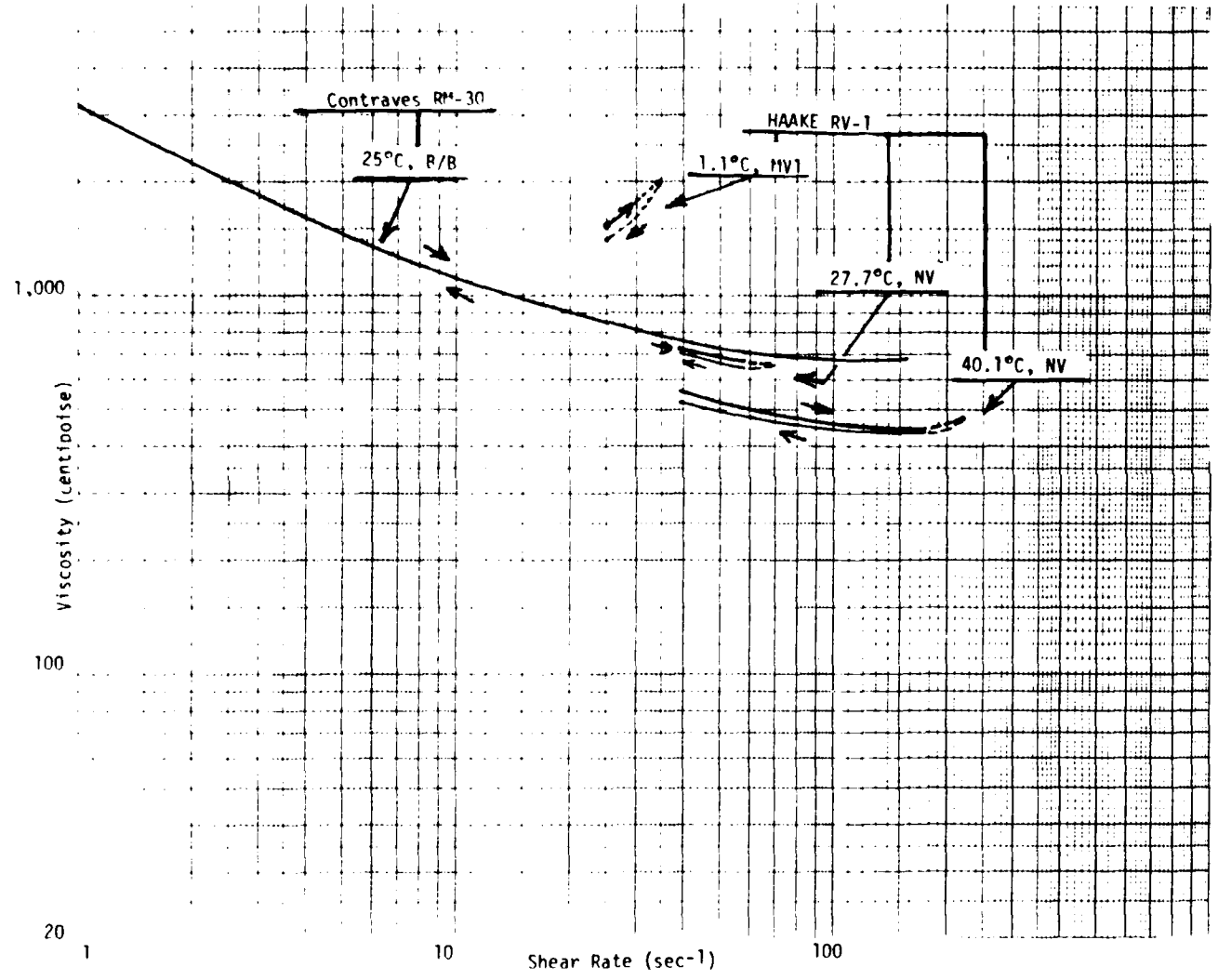



Figure 10. Viscosity Profile of Carbon Slurry 3856-64 (60 wt.% of SL-90)

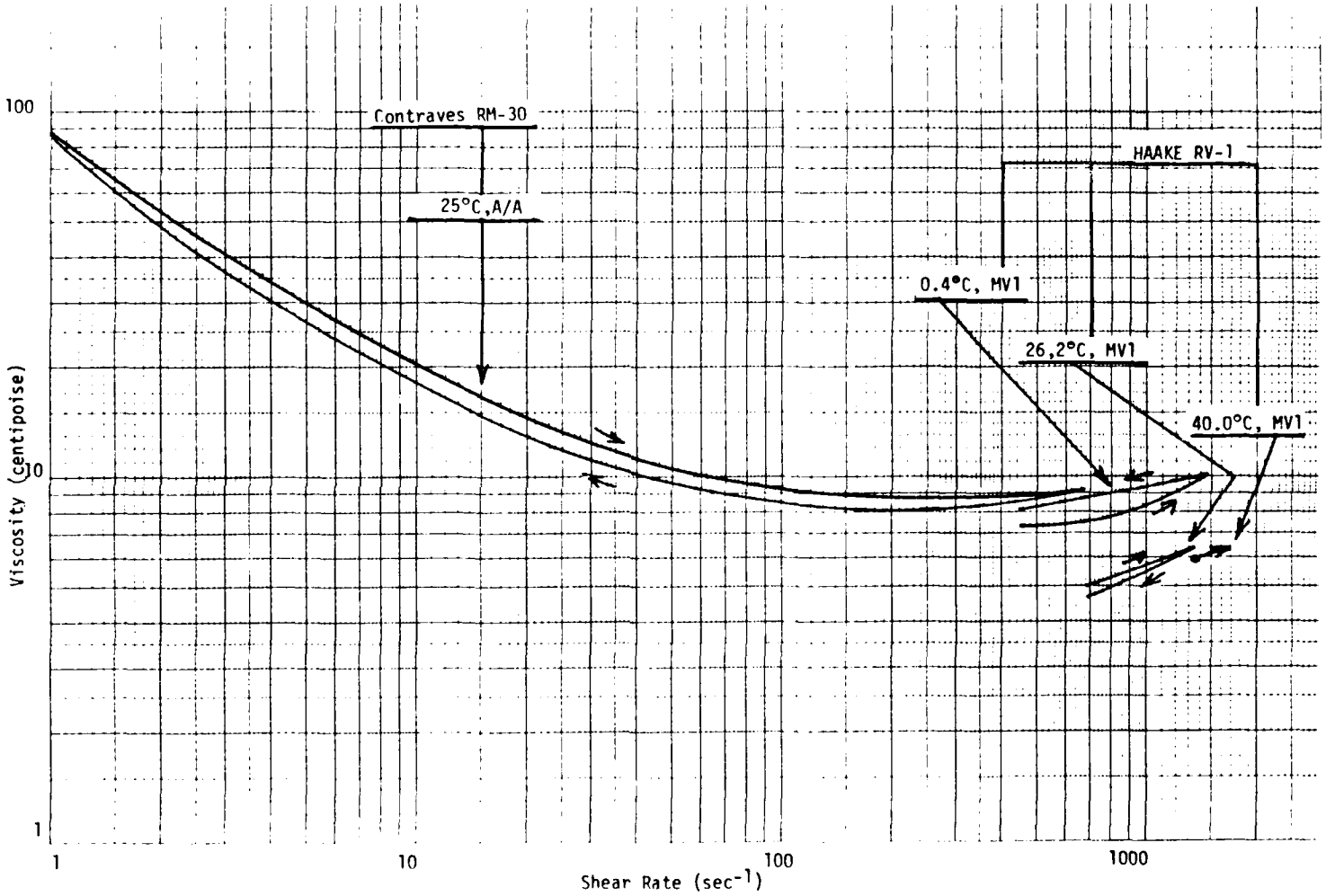



Figure 11. Viscosity Profile of Carbon Slurry 3856-123 (25 wt.% of Statex MT)

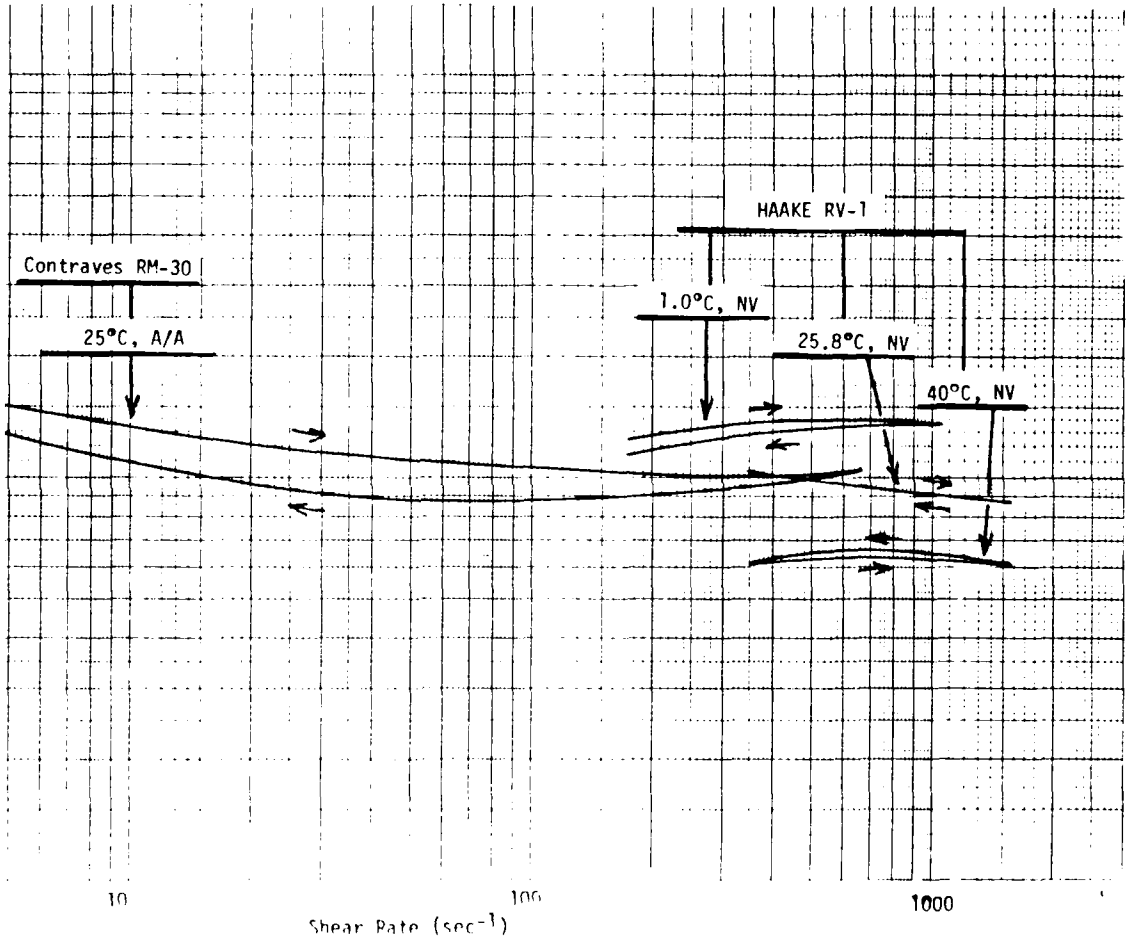



Figure 12. Viscosity Profile of Carbon Slurry 3856-83
(25 wt. % of SL-90)

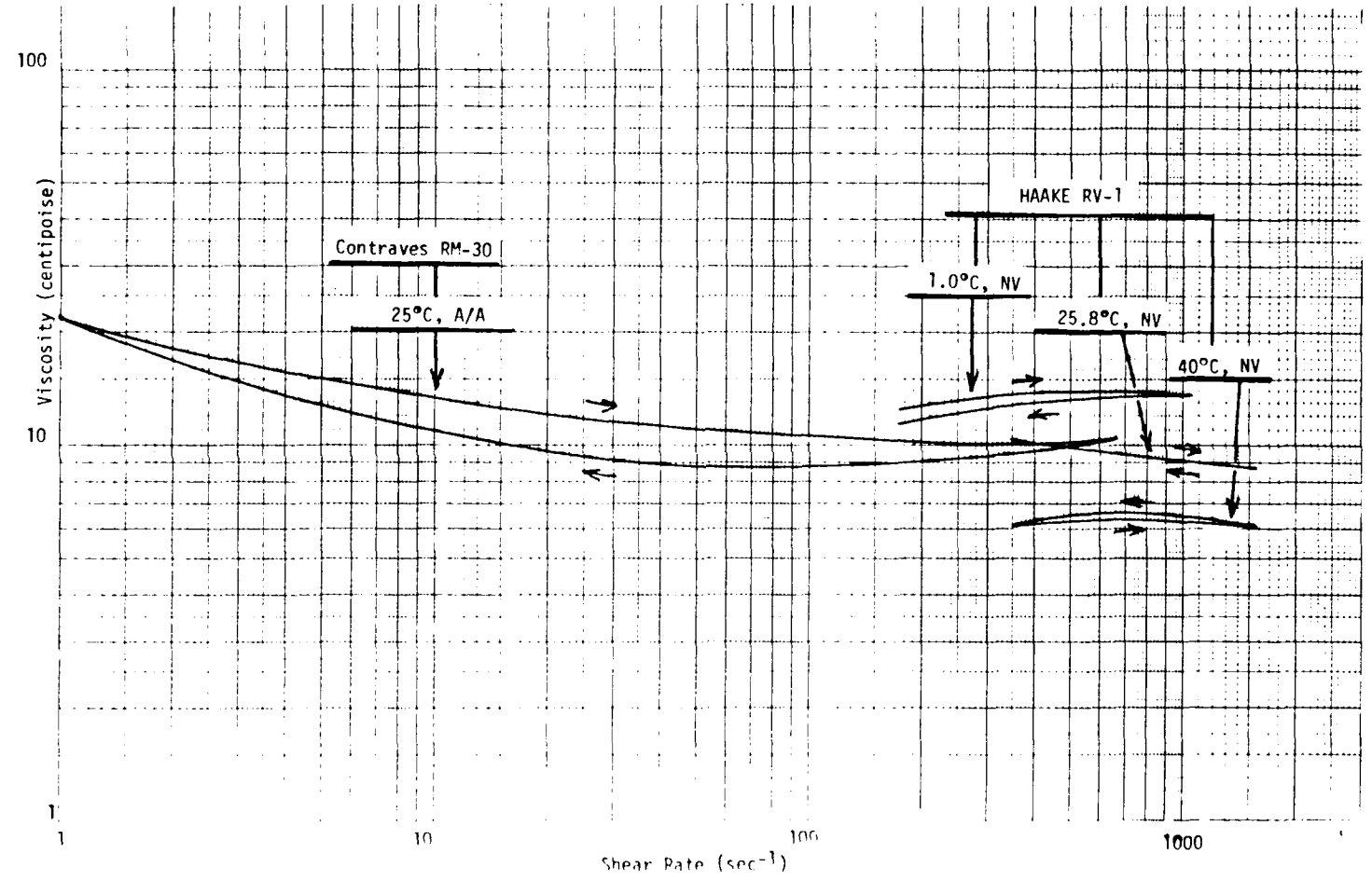



Figure 12. Viscosity Profile of Carbon Slurry 3856-83
(25 wt. % of SL-90)

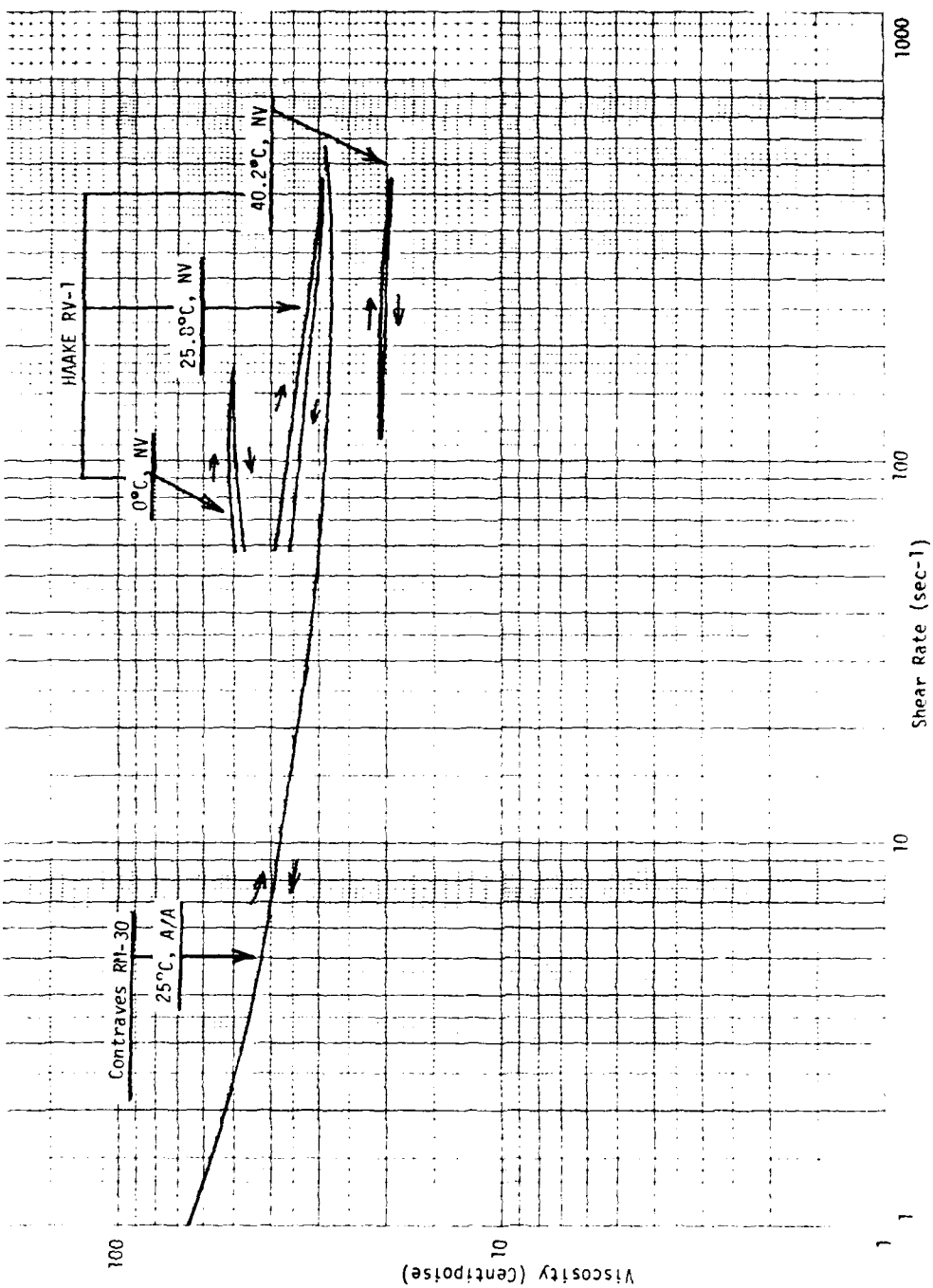



Figure 13. Viscosity Profile of Carbon Slurry 3856-77 (25 wt.% of N-660)

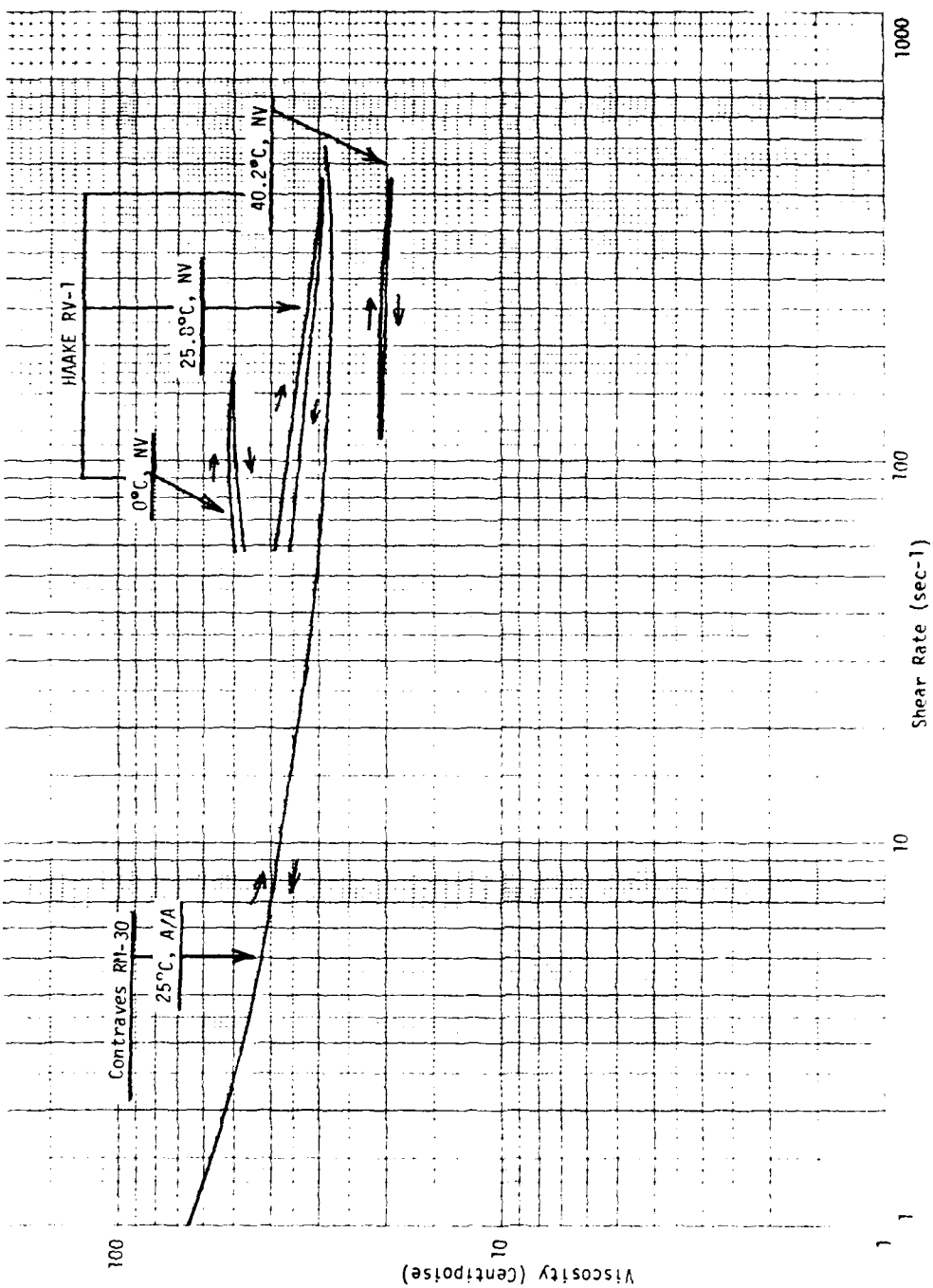



Figure 13. Viscosity Profile of Carbon Slurry 3856-77 (25 wt.% of N-660)

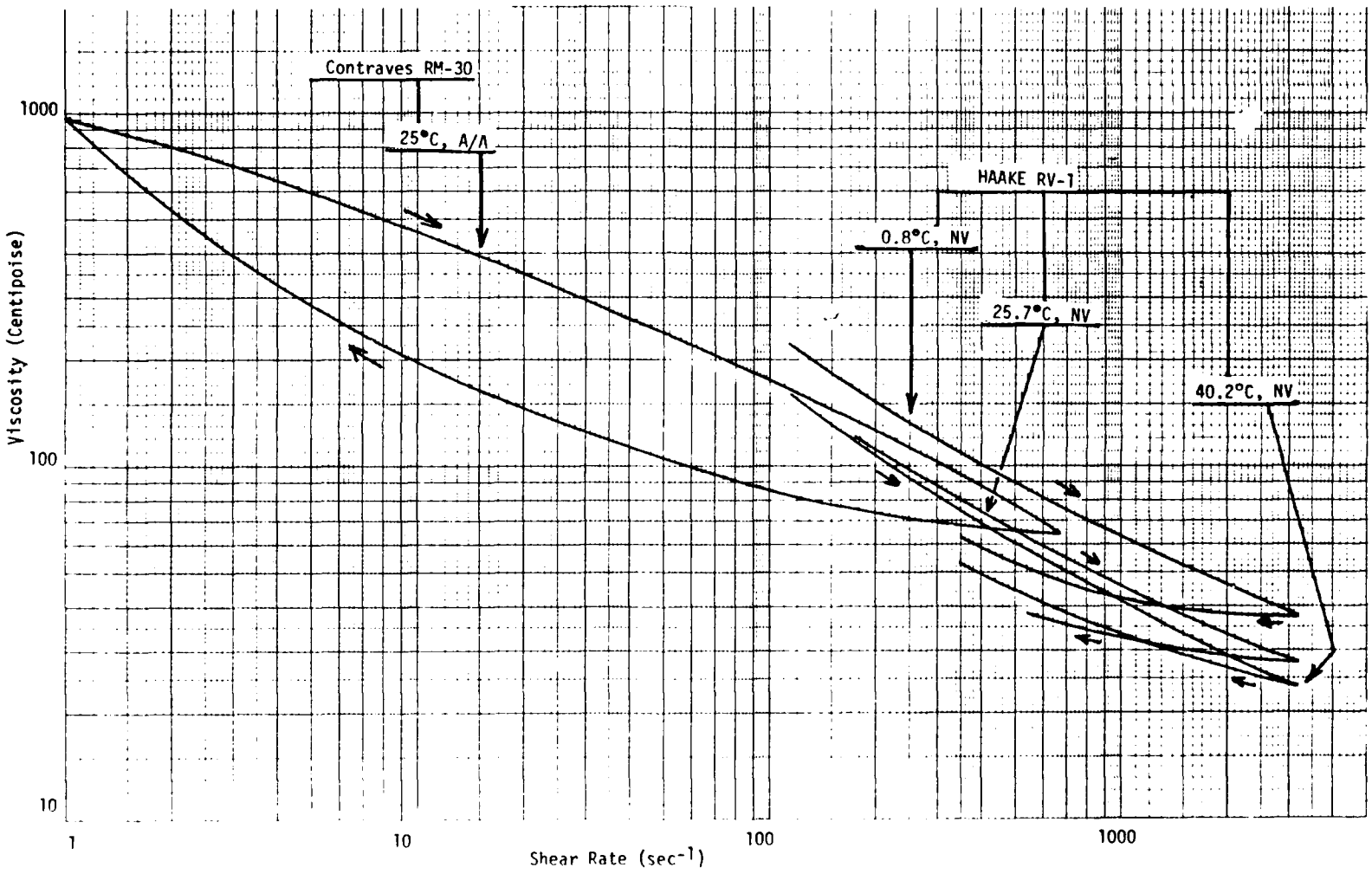



Figure 14. Viscosity Profile of Carbon Slurry 3856-99 (25 wt.% of N-219)

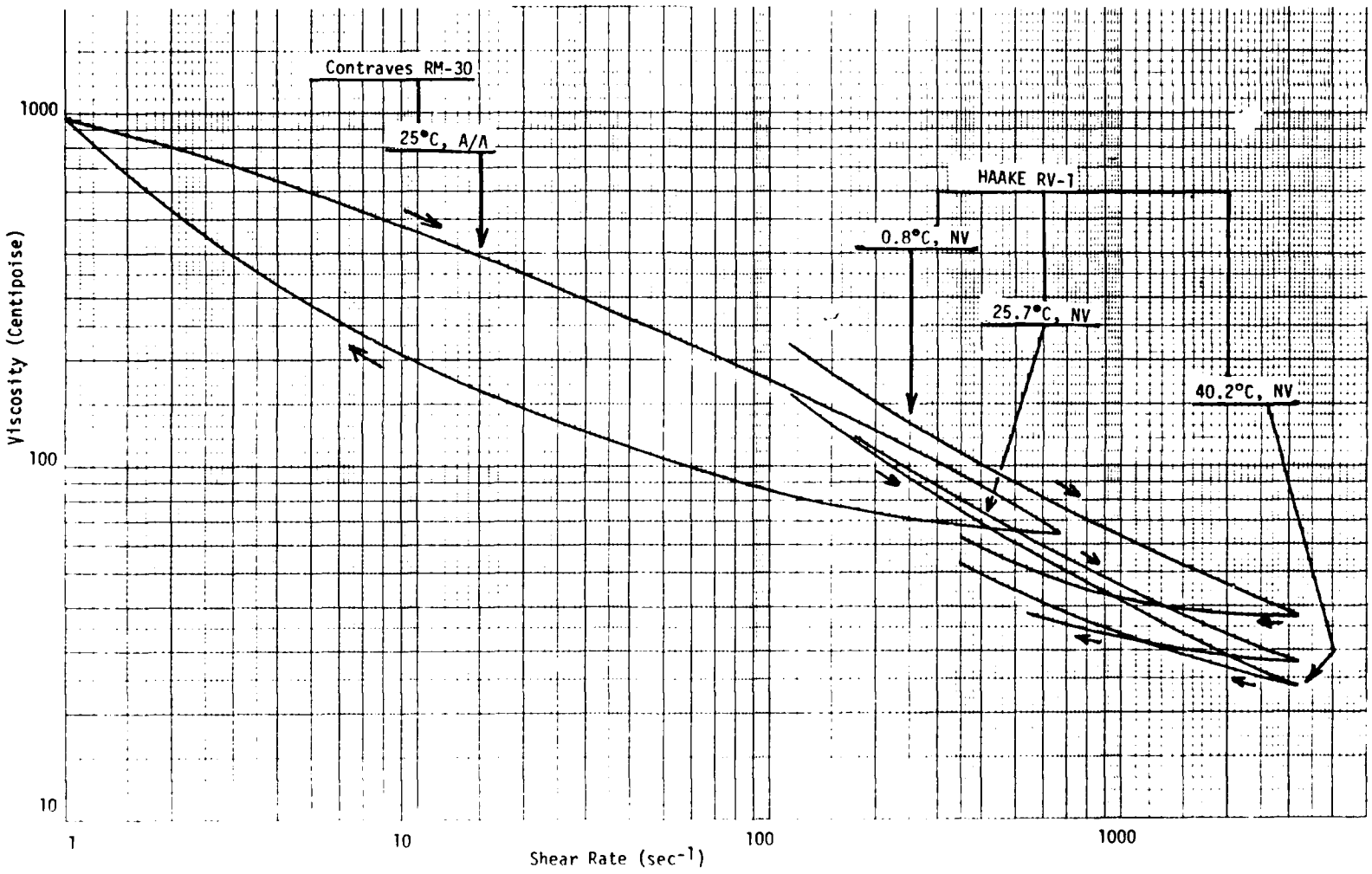



Figure 14. Viscosity Profile of Carbon Slurry 3856-99 (25 wt.% of N-219)

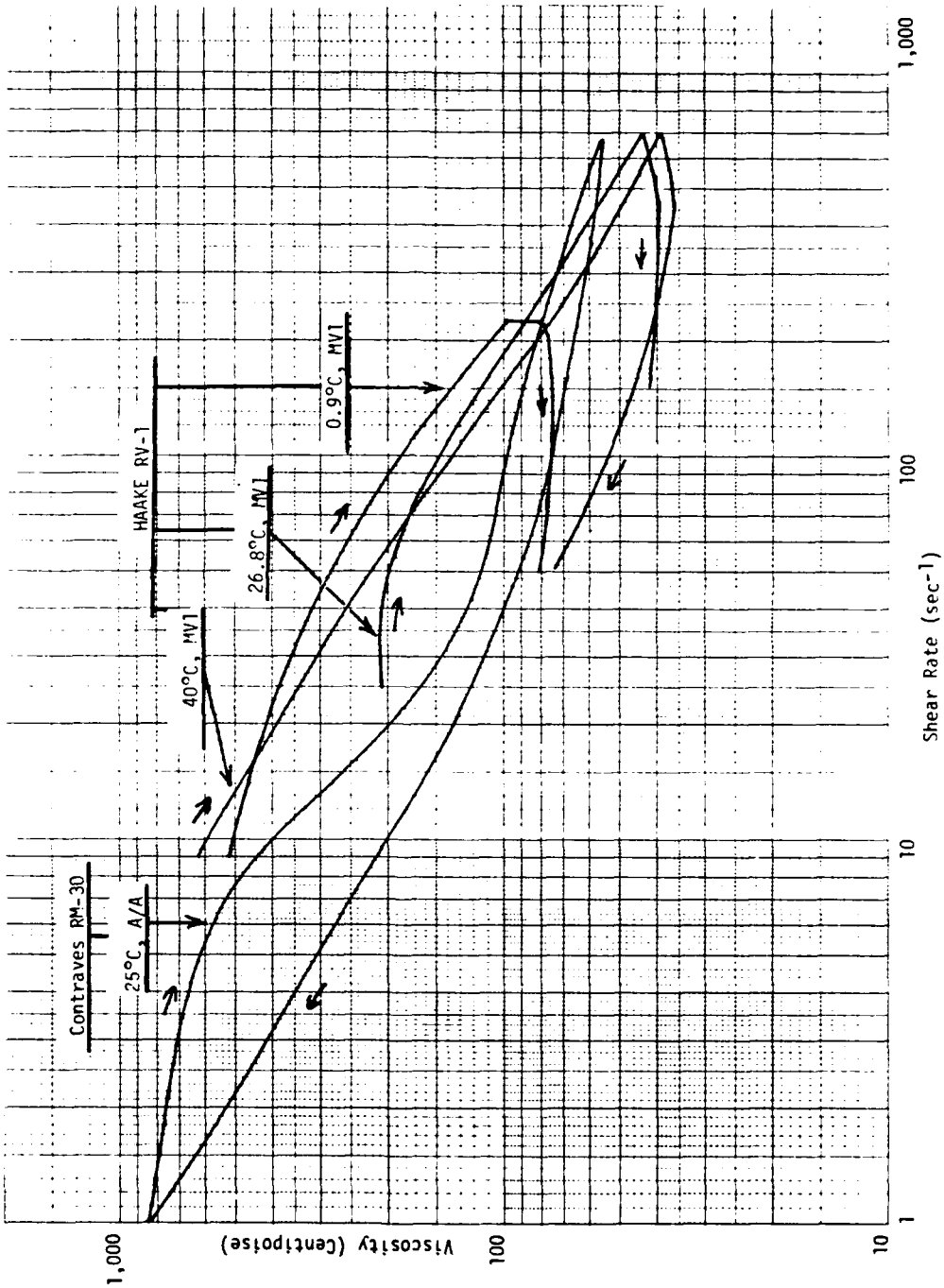



Figure 15. Viscosity Profile of Carbon Slurry 3908-21 (25 wt.% of CBPP-5)

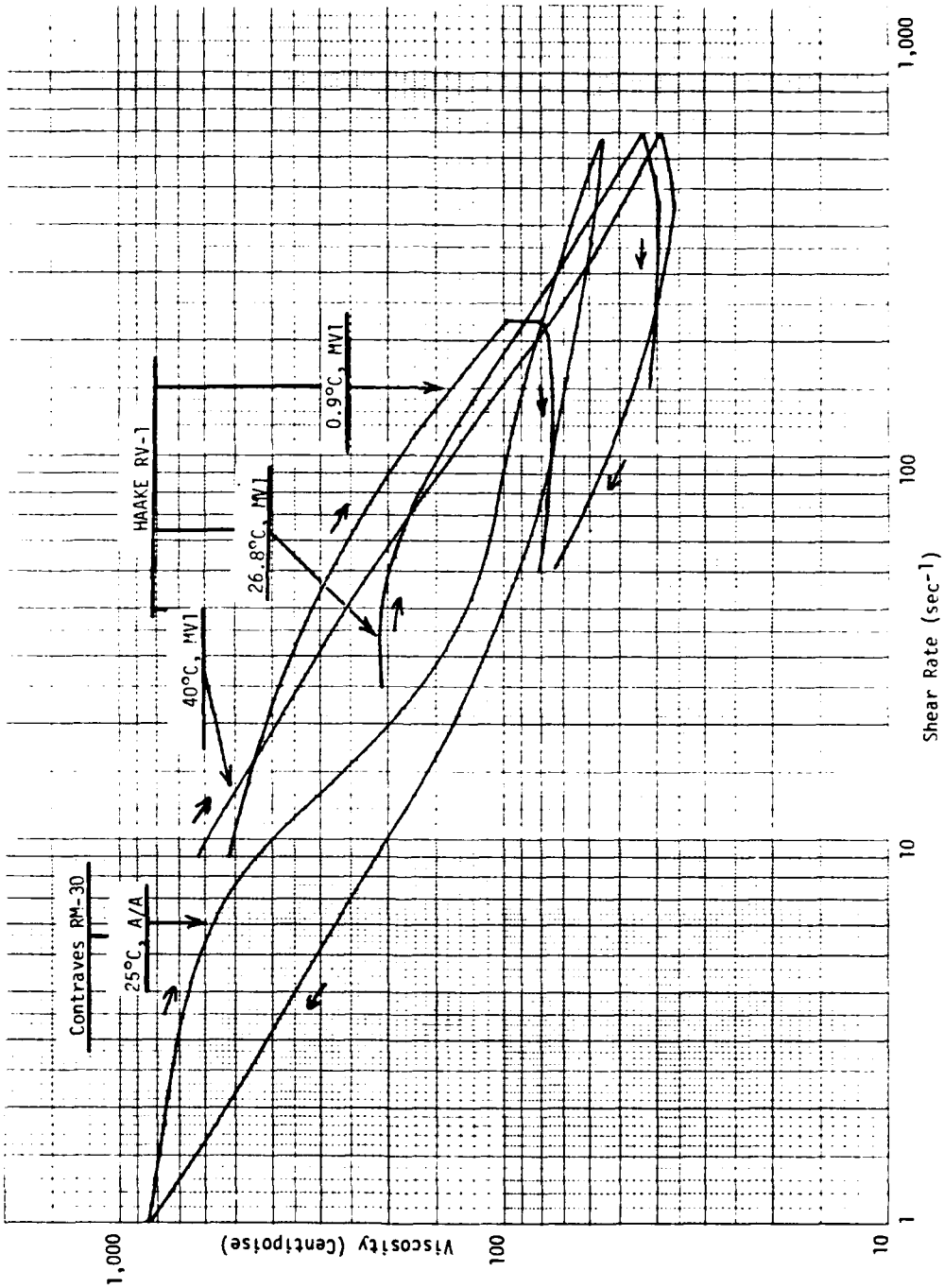



Figure 15. Viscosity Profile of Carbon Slurry 3908-21 (25 wt.% of CBPP-5)

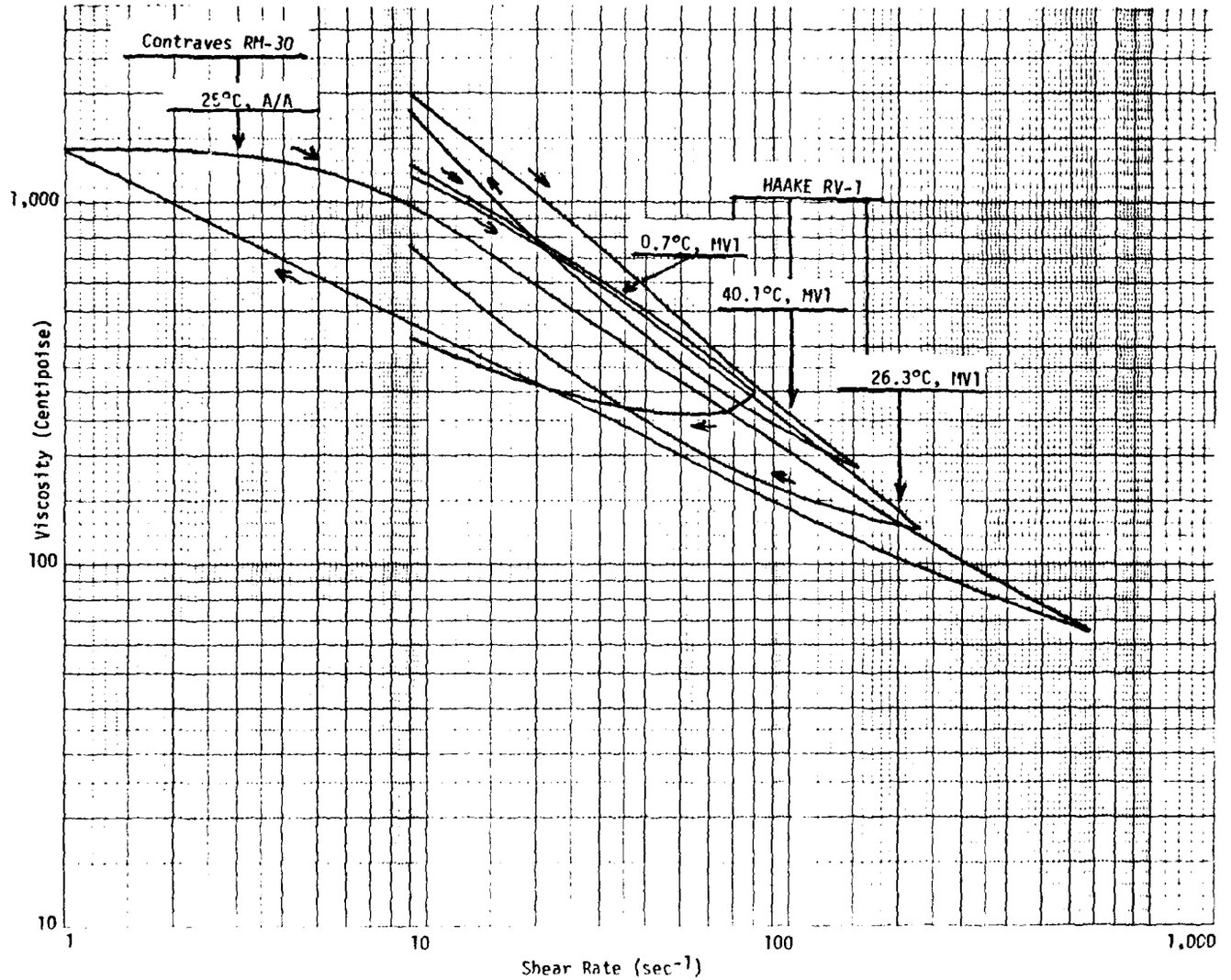



Figure 16. Viscosity Profile of Carbon Slurry 3908-22
(25 wt.% of CBPP-12-1, containing 0.21% Fe)

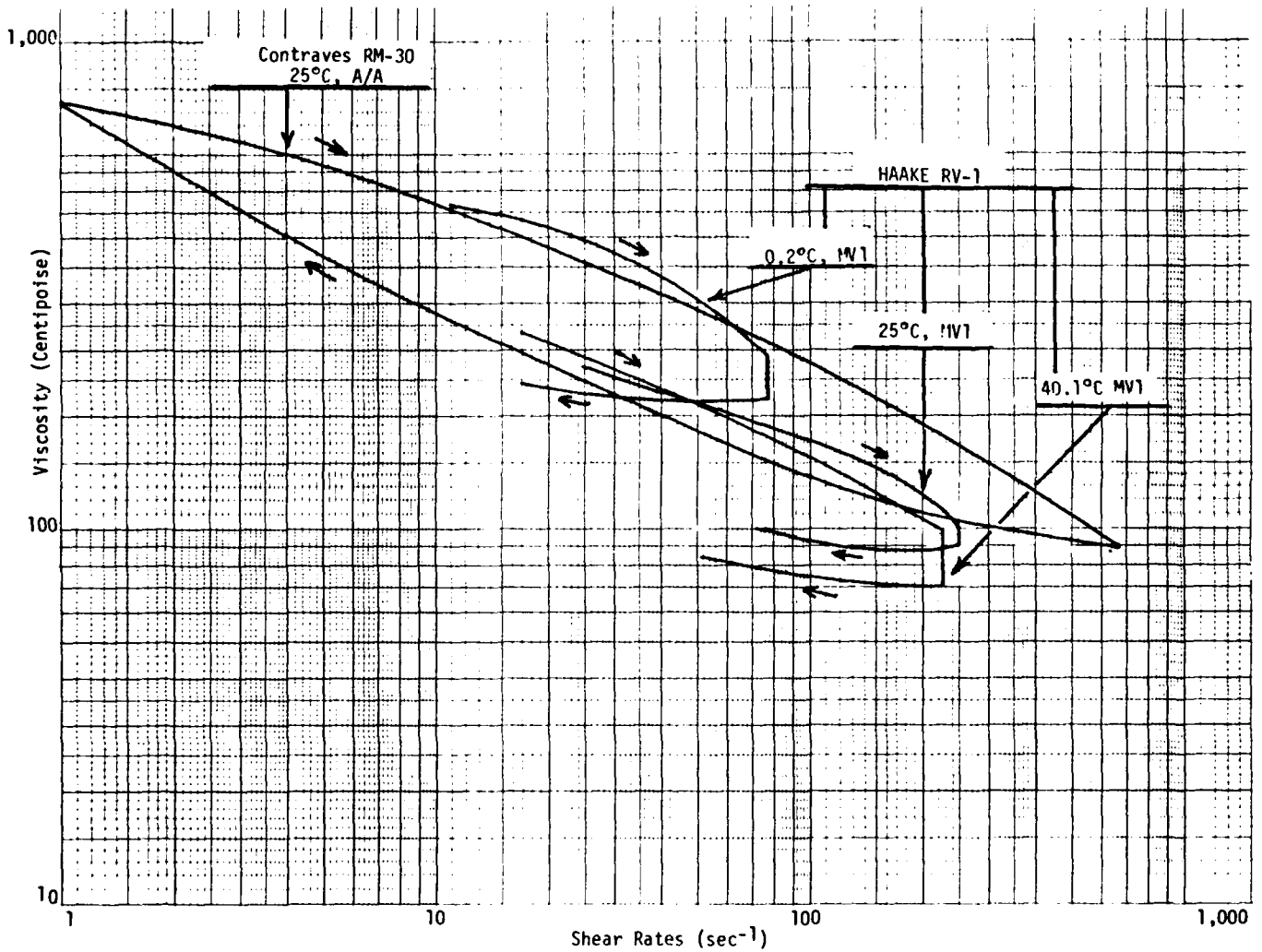



Figure 17. Viscosity Profile of Carbon Slurry 3908-9
(25 wt.% of CBPP-10-1, containing 0.99% Fe)

III. C.

4. Delivery of Carbon Slurry Fuels

A total of nine carbon slurries (5 gallons each) have been prepared and characterized. Three gallons of each slurry have been sent to J. R. McCoy at Wright-Patterson Air Force Base for physical evaluation. Six samples were sent in July 1980 and three in August 1980. Two gallons of each carbon slurry have been sent to T. W. Bruce at AiResearch Manufacturing Company of Arizona in August 1980. A summary of the carbon slurries is given below.

| Slurry Ref. No. | Type of Carbon Black | Average Particle Diameter (nm) | Weight % Carbon Black | W-P AFB (3 gal) | AiResearch (2 gal) |
|---|---|---|---|---|---|
| 3856-41 | N990 (MT) | 300 | 65 | July | August |
| 3856-64 | SL-90 | 103 | 60 | July | August |
| 3856-123 | N990 (MT) | 300 | 25 | July | August |
| 3856-83 | SL-90 | 103 | 25 | July | August |
| 3856-77 | N660 | 56 | 25 | July | August |
| 3856-99 | N219 | 30 | 25 | July | August |
| 3908-21 | Control CB | 37 | 25 | August | August |
| 3908-22 | 0.21% Cat/CB | 37 | 25 | August | August |
| 3908-9 | 0.99% Cat/CB | 26 | 25 | August | August |

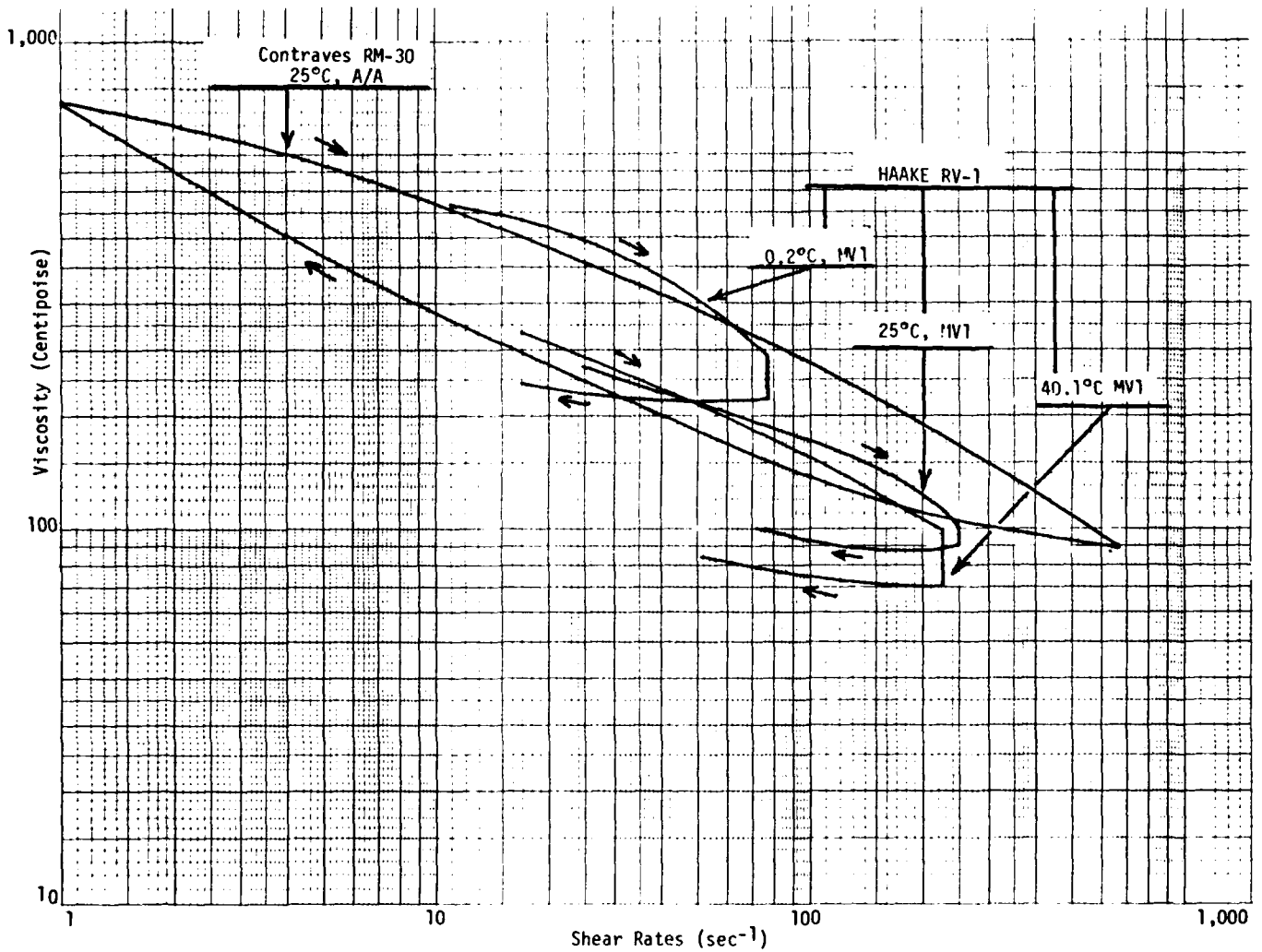



Figure 17. Viscosity Profile of Carbon Slurry 3908-9
(25 wt.% of CBPP-10-1, containing 0.99% Fe)

III.

D. Combustion Testing of Carbon Slurry Fuels

The combustion tests for the program are to be conducted by AiResearch Manufacturing Company of Arizona. Unexpected delays have set back the combustion testing of the nine carbon slurry fuel samples. It is anticipated that some of the samples will be tested in February 1981.

The samples will be evaluated in a modified version of the atmospheric combustor rig used by AiResearch in 1979 to conduct combustion tests on carbon slurry fuels provided by Suntech (Reference 8).

IV. Recommendations

In order to enhance the formulation and combustion characteristics of carbon slurry fuels, Ashland recommends a carbon slurry fuel development program that would encompass the following:

1. Thoroughly investigate the influence that fundamental *carbon black properties*, such as particle size, aggregate size, structure and surface area have on the formulation and combustion behavior of carbon slurry fuels.

2. Evaluate the effect various carbon blacks containing potential combustion catalysts such as cobalt, manganese, nickel and lead have on the formulation and combustion behavior of carbon slurry fuels.

3. Perform the combustion studies in a laboratory combustor with which it is possible to accurately measure the rate of carbon burn out.

4. Evaluate those slurries which are found to have enhanced carbon burn out rates in a can-type or a small turbine engine combustor.

5. Prepare production size quantities of the best carbon slurry fuels incorporating the most active combustion catalyst with the best slurry formulation.

6. Evaluate the combustion behavior of those fuels produced in #5 above in a turbine engine.

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| 3856-64 | SL-90 | 103 | 60 | July | August |
| 3856-123 | N990 (MT) | 300 | 25 | July | August |
| 3856-83 | SL-90 | 103 | 25 | July | August |
| 3856-77 | N660 | 56 | 25 | July | August |
| 3856-99 | N219 | 30 | 25 | July | August |
| 3908-21 | Control CB | 37 | 25 | August | August |
| 3908-22 | 0.21% Cat/CB | 37 | 25 | August | August |
| 3908-9 | 0.99% Cat/CB | 26 | 25 | August | August |

## References


1) R. H. Salvesen, Carbon Slurry Fuels for Volume Limited Missiles, Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio 45433, AFAPL-TR-79-2122.

2) T. W. Bruce, H. C. Mongia, R. S. Stearns, L. W. Hall, E. M. Faeth, Formulation Properties, and Combustion of Carbon-Slurry Fuels, 16th JANNAF Combustion Meeting, Vol. III, CPIA publication 308, Dec. 1979, p. 679.

3) R. H. Salvesen, Carbon Slurry Fuels, Monthly Report No. 20, June 3, 1980, Air Force Contract No. F33615-78-C-2025, Exxon Research and Engineering Company.

4) J. B. Howard, W. J. Kausch, Jr., Soot Control by Fuel Additives - A Review, Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall Air Force Base, Florida 32403, Final Report, Sept. 1979

5) P. L. Walker, Jr., M. Shelef, R. A. Anderson, Catalysis of Carbon Gasification in "Chemistry and Physics of Carbon" Vol. 4, p. 287, P. L. Walker, Jr., Ed. 1968.

6) R. H. Essenhigh, Combustion and Flame Propagation in Coal Systems: A Review, in 15th Symposium (International) on Combustion, p. 353-372; the Combustion Institute, Pittsburgh, Pa.(1977)

7) R. H. Salvesen, Carbon Slurry Fuels, Monthly Report No. 24, Oct. 3, 1980, Air Force Contract No. F33615-78-C-2025, Exxon Research and Engineering Company.

8) T. W. Bruce, H. Mongia, Compound Cycle Turbofan Engine Task IX, Carbon Slurry Fuel Combustion Evaluation Program, Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio 45433, Technical Report AFWAL-TR-80-2035, March 1980.

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6. Evaluate the combustion behavior of those fuels produced in #5 above in a turbine engine.

## APPENDIX A

CARBON BLACK PROPERTY TESTS:

1. Carbon Black - Iodine Adsorption Number

   This test is standardized in ANSI/ASTM procedure D 1510-76. The iodine number of a certain carbon black, which is usually given in mg/g or mg/100g, is a measurement of the surface area of carbon black particles per unit weight. It correlates well with BET (nitrogen adsorption measurements) for furnace blacks, but it is a much quicker test.

2. Carbon Black - Dibutyl Phthalate Absorption Number

   This test is standardized in ANSI/ASTM procedure D 2414-76. The DBP number, which is usually given in $cm^3/g$ or $cm^3/100g$, is the principal measurement of a black's structure (the degree of interlinkage between particles). A high DBP number indicates a greater structure.

3. Carbon Black - Tint Strength

   This test is standardized in ANSI/ASTM procedure D 3265-76. The tint test is a reflectance test which measures the relative ability of a black to hide a white vehicle. The ASTM tint values are highly dependent upon the particle size and the structure of the black. These values are based on the ASTM tint reference black and are reported as the percent reflectance, as compared to this reference black.

4. Carbon Black Extractables - Toluene Discoloration

   This test is standardized in ANSI/ASTM procedure D 1618-75. In the toluene stain test, the black is extracted with toluene. This toluene is then checked for discoloration and the toluene stain is the percent transmittance at 425 nm wavelength measured by a spectrophotometer. This test is a measure of how much oil is on the black.

5. Carbon Black - Heating Loss

   This test is standardized in ASTM procedure D 1509-75. The heat loss of a black indicates the weight percent of moisture absorbed by the black.

6. Carbon Black - Particle Size

   A Phillips Transmission Electron Microscope (Model #EM-200) is used to measure the particle size distributions of the carbon blacks.

## References


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The black sample is dispersed in a solvent and the dispersion is deposited on a microscope grid. After the solvent evaporates, the sample is inserted into the TEM and photographed at about 5,000x to 10,000x. After photographic development, the negatives are enlarged to 8" x 10" transparencies, corresponding to a final enlargement of 100,000x. A Zeiss Particle Size Counter is employed for particle size "counting" and particle size distributions are plotted using a programmable calculator.

A simplified method for determining the Calculated Average Particle Diameter using only the tint and DBP absorption number was developed by Dr. Norman Smith at Ashland Chemical Company. By means of an extensive electron microscope study in combination with DBP absorptive and tint data, an equation was derived which would express a carbon blacks' particle size as a function of its tint and DBP absorption. The equation is given below:

$$\text{Particle Size (nm)} = 45.6 - 88.72(\text{DBP}) + 35.95\,(\text{DBP})^2 + \frac{36.77}{(\text{Tint}/100)}$$

DBP units = $cm^3/gram$
Tint units = percent of IRB #3

7. Carbon Black - Surface Area

A Quantachrome Corp. Quantasorb is employed to measure the surface area of the carbon black. This instrument measures the amount of nitrogen adsorbed by the carbon black sample. Since sample weight, nitrogen partial pressure, temperature, and amount adsorbed are known, the BET (Brunauer-Emmett-Teller) equation is then used to calculate the surface area of the black. Routinely, three adsorption points are taken at 10%, 20%, and 30% nitrogen in helium to give a BET surface area. Three samples are usually tested, of which the mean average is taken as the reported BET surface area. The BET surface areas are reported in $m^2/gm$.

8. Carbon Black - Metal Analysis

An Instrumentation Laboratories, Inc. Atomic Absorption-Emission Spectrophotometer (Model 353) is used to analyze the amount of metal catalyst incorporated in the carbon black. The black samples are ashed in a furnace and the ash is then digested with 10 ml. of 6N HCl. The residue is then diluted with 100 ml of $H_2O$. This solution is then analyzed with the Spectrophotometer, which is calibrated with solutions of known metal concentrations.

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5. Carbon Black - Heating Loss

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6. Carbon Black - Particle Size

   A Phillips Transmission Electron Microscope (Model #EM-200) is used to measure the particle size distributions of the carbon blacks.

APPENDIX B

CARBON SLURRY PROPERTY TESTS:

1. Carbon Slurry - Density @25°C

The density of a slurry is measured with a Gardo Pyncnometer cup. The slurry is kept in a 25°C temperature bath until the slurry temperature comes to equilibrium. Then the cup is filled with 83.2cc of slurry. The density is calculated from the difference in weight between a full and an empty cup.

2. Carbon Slurry - Heat of Combustion

A Parr Automatic Bomb Calorimeter (Model 1241) with a Parr 1680 Master Control is used to measure the heat of combustion of the carbon slurries. This test is standardized in the ANSI/ASTM D 240-76 procedure. From this instrument the net heat content of each slurry is obtained. By multiplying the net heat content by the density, the net volumetric heat content is obtained.

3. Carbon Slurry - Brookfield Viscosity

The Brookfield viscosity of the slurries is measured with a Brookfield Synchro-Lectric RVT Viscometer. The slurry is poured into a 600 ml. beaker at a temperature of 25°C. One of seven spindles is chosen for measuring the slurry viscosity and the instrument is operated under normal Brookfield procedures. The Brookfield viscosity is measured at 100, 50, and 20 RPM. The maximum shear rate of this instrument is 10 $sec^{-1}$.

4. Carbon Slurry - Absolute Viscosity

The absolute viscosity profiles of the slurries are measured with a Haake Rotovisco RV-1, or a Contraves Rheomat RM-30 viscometer. Both of these instruments utilize the cup and bob measuring systems.

The Haake RV-1 has two interchangeable measuring heads; the MK 50 head has a torque range of 0 to 50 gram-centimeters and the MK 500 head has a torque range of 0 to 500 gram-centimeters. The higher torque range allows the measurement of higher shear stress values and thus higher viscosities. The NV measuring system is primarily for low viscosity liquids. It is a double gap cylinder system consisting of a bell shaped rotor and a cup. The MV measuring system consists of a cup and three rotors of different diameter. This system is primarily for medium viscosity liquids. Characteristics of these measuring systems are given in the following table.

The black sample is dispersed in a solvent and the dispersion is deposited on a microscope grid. After the solvent evaporates, the sample is inserted into the TEM and photographed at about 5,000x to 10,000x. After photographic development, the negatives are enlarged to 8" x 10" transparencies, corresponding to a final enlargement of 100,000x. A Zeiss Particle Size Counter is employed for particle size "counting" and particle size distributions are plotted using a programmable calculator.

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The Contraves RM-30 has four torque ranges of 0 to 50, 100, 200, and 500 gram-centimeters. The instrument has the capability of scanning a given shear rate range at a programmed rate and plotting the shear stress values on an X-Y recorder. The measuring systems designated as MS A/A, MS B/B, MS C/C, MS D/DE, and MS E/DE all consist of a bob and cup. The characteristics of these measuring systems are given in the table below.

Viscosity Measuring Systems

| Measuring System | Maximum Viscosity (cp) | Maximum Shear Rate ($sec^{-1}$) | Cylinder Wall Gap (mm) | Sample Volume ($cm^3$) |
|---|---|---|---|---|
| Haake | | | | |
| NV | $10^4$ | 5400 | 0.35 | 9 |
| MV I | $10^5$ | 2300 | 0.96 | 40 |
| MV II | $10^6$ | 900 | 2.6 | 55 |
| MV III | $10^7$ | 440 | 5.8 | 70 |
| Contraves | | | | |
| MS A/A | $10^6$ | 660 | 1.30 | 120 |
| MS B/B | $10^7$ | 155 | 4.00 | 80 |
| MS C/C | $10^8$ | 95 | 3.20 | 20 |
| MS D/DE | $10^9$ | 55 | 3.75 | 8 |
| MS E/DE | $10^{10}$ | 28 | 5.50 | 9 |

5. Carbon Slurry - Stability Test

The stability of the carbon slurry is tested by centrifuging a 75 ml. slurry sample for 30 minutes at a force of 900g. The DAMON/IEC Model # centrifuge is used for this test. After centrifugation, the sample is inspected with probes for carbon packing and separation. The slurry is then poured out, and the residue left in the tube is inspected. If there is no evidence of separation or carbon packing, the sample passes the test. The test can be varied by subjecting the sample to a longer centrifugation time and/or by increasing the centrifugation force.

6. Carbon Slurry - Flash Point

The flash point of a carbon slurry is measured with a Setaflash tester. The normal flash point determination procedure is followed, as described in ASTM method D3243.

APPENDIX B

CARBON SLURRY PROPERTY TESTS:

1. Carbon Slurry - Density @25°C

The density of a slurry is measured with a Gardo Pyncnometer cup. The slurry is kept in a 25°C temperature bath until the slurry temperature comes to equilibrium. Then the cup is filled with 83.2cc of slurry. The density is calculated from the difference in weight between a full and an empty cup.

2. Carbon Slurry - Heat of Combustion

A Parr Automatic Bomb Calorimeter (Model 1241) with a Parr 1680 Master Control is used to measure the heat of combustion of the carbon slurries. This test is standardized in the ANSI/ASTM D 240-76 procedure. From this instrument the net heat content of each slurry is obtained. By multiplying the net heat content by the density, the net volumetric heat content is obtained.

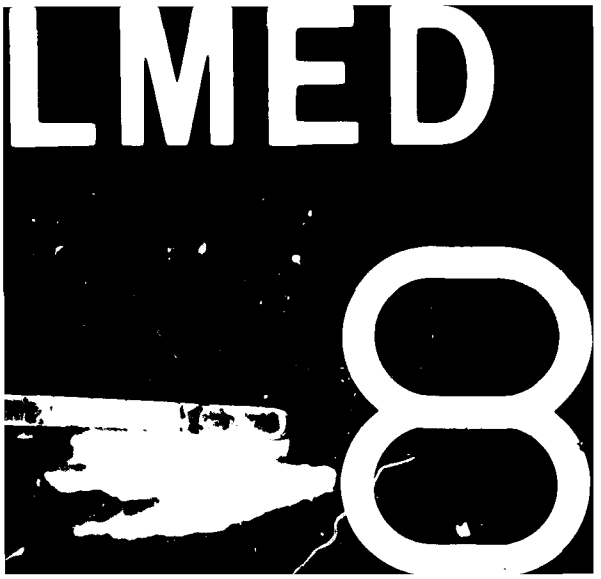
3. Carbon Slurry - Brookfield Viscosity

The Brookfield viscosity of the slurries is measured with a Brookfield Synchro-Lectric RVT Viscometer. The slurry is poured into a 600 ml. beaker at a temperature of 25°C. One of seven spindles is chosen for measuring the slurry viscosity and the instrument is operated under normal Brookfield procedures. The Brookfield viscosity is measured at 100, 50, and 20 RPM. The maximum shear rate of this instrument is 10 $sec^{-1}$.

4. Carbon Slurry - Absolute Viscosity

The absolute viscosity profiles of the slurries are measured with a Haake Rotovisco RV-1, or a Contraves Rheomat RM-30 viscometer. Both of these instruments utilize the cup and bob measuring systems.

The Haake RV-1 has two interchangeable measuring heads; the MK 50 head has a torque range of 0 to 50 gram-centimeters and the MK 500 head has a torque range of 0 to 500 gram-centimeters. The higher torque range allows the measurement of higher shear stress values and thus higher viscosities. The NV measuring system is primarily for low viscosity liquids. It is a double gap cylinder system consisting of a bell shaped rotor and a cup. The MV measuring system consists of a cup and three rotors of different diameter. This system is primarily for medium viscosity liquids. Characteristics of these measuring systems are given in the following table.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The Contraves RM-30 has four torque ranges of 0 to 50, 100, 200, and 500 gram-centimeters. The instrument has the capability of scanning a given shear rate range at a programmed rate and plotting the shear stress values on an X-Y recorder. The measuring systems designated as MS A/A, MS B/B, MS C/C, MS D/DE, and MS E/DE all consist of a bob and cup. The characteristics of these measuring systems are given in the table below.

Viscosity Measuring Systems

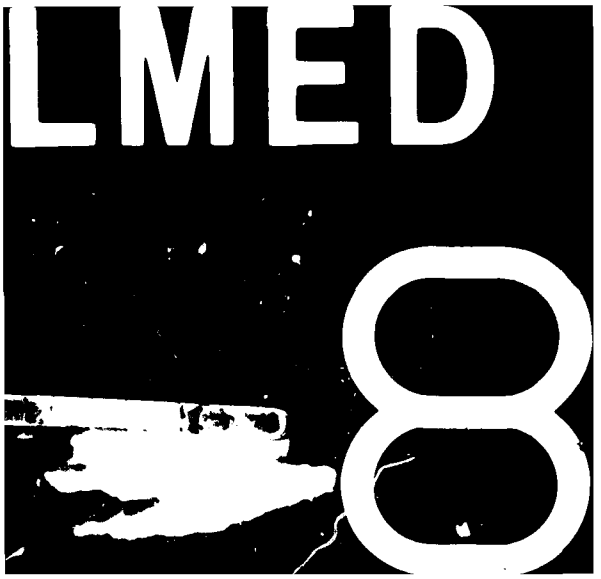
| Measuring System | Maximum Viscosity (cp) | Maximum Shear Rate ($sec^{-1}$) | Cylinder Wall Gap (mm) | Sample Volume ($cm^3$) |
|---|---|---|---|---|
| Haake | | | | |
| NV | $10^4$ | 5400 | 0.35 | 9 |
| MV I | $10^5$ | 2300 | 0.96 | 40 |
| MV II | $10^6$ | 900 | 2.6 | 55 |
| MV III | $10^7$ | 440 | 5.8 | 70 |
| Contraves | | | | |
| MS A/A | $10^6$ | 660 | 1.30 | 120 |
| MS B/B | $10^7$ | 155 | 4.00 | 80 |
| MS C/C | $10^8$ | 95 | 3.20 | 20 |
| MS D/DE | $10^9$ | 55 | 3.75 | 8 |
| MS E/DE | $10^{10}$ | 28 | 5.50 | 9 |

5. Carbon Slurry - Stability Test

The stability of the carbon slurry is tested by centrifuging a 75 ml. slurry sample for 30 minutes at a force of 900g. The DAMON/IEC Model # centrifuge is used for this test. After centrifugation, the sample is inspected with probes for carbon packing and separation. The slurry is then poured out, and the residue left in the tube is inspected. If there is no evidence of separation or carbon packing, the sample passes the test. The test can be varied by subjecting the sample to a longer centrifugation time and/or by increasing the centrifugation force.

6. Carbon Slurry - Flash Point

The flash point of a carbon slurry is measured with a Setaflash tester. The normal flash point determination procedure is followed, as described in ASTM method D3243.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